PB89-167910

Technology Evaluation Report
SITE (Superfund Innovative Technology Evaluation)
Program Demonstration Test Shirco Pilot-Scale
Infrared Incineration System at the
Rose Township Demode Road Superfund Site. Volume 2

Enviresponse, Inc., Livingston, NJ

Prepared for:

Environmental Protection Agency, Cincinnati, OH

Apr 89

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16. ABSTRACT

The performance of the Shirco pilot-scale infrared thermal destruction system has been evaluated at the Rose Township, Demode Road Superfund Site and is presented in the report. The waste tested consisted of solvents, organics and heavy metals in an illegal dump site.

Volume I gives the performance results, summary of data and tests performed along with the process description.

Volume II is the test program, copies of the original operating data, sampling analysis report and quality assurance plan.

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TECHNOLOGY EVALUATION REPORT:
SITE Program Demonstration Test
Shirco Pilot-Scale Infrared Incineration System at the Rose Township Demode Road Superfund Site

Volume II

RISK REDUCTION ENGINEERING LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OH 45268

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VOLUME II

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* Volume I contains the Shirco Rose Township process description, field operations documentation, performance data evaluation, summary of sampling and analysis program, and an economic analysis.

APPENDIX A

OPERATING LOG DATA

The operating log data consists of 56 pages of handwritten operations data log sheets and comments. If the data is required for further analysis of this report, please contact Mr. H. Wall, SITE Project Manager, USEPA, Waste Minimization Destruction and Disposal Research Division, Risk Reduction Engineering Laboratory, Cincinnati, Ohio.

A comprehensive summary of the operations data is presented in Tables 7 and 8, Volume I, Section 4.

APPENDIX B

SAMPLING AND ANALYTICAL REPORT

RADIAN
CORPORATION

DCN 88-274-013-03

SAMPLING AND ANALYTICAL CHARACTERIZATION OF THE SHIRCO INFRARED INCINERATION SYSTEM AT THE ROSE TOWNSHIP HAZARDOUS WASTE SITE

Final Report

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1.0 INTRODUCTION AND OBJECTIVES

In 1979, the U. S. Environmental Protection Agency (U.S. EPA) designated a dump site near Flint, Michigan, the Rose Township-Demode Road site, as a hazardous waste site, and it became eligible for funding under the Superfund program. It was further selected as a demonstration site to test on-site incineration as one of the emerging clean-up technologies under the SITE (Superfund Innovative Technologies Evaluation) program. The U.S. EPA's Office of Solid Waste and Emergency Response (OSWER) contracted with Enviresponse, Inc. of Edison, N.J. (Enviresponse) to manage the demonstration and Shirco Infrared Systems of Dallas, Texas (Shirco) provided the incineration system. Enviresponse subsequently engaged Radian Corporation of Austin, Texas (Radian) to provide sampling and analytical services during the test incineration. This document is Radian's report to Enviresponse describing the sampling and analytical program and giving the results.

This report is not a comprehensive description of incineration work done at the Rose Township-Demode Road site. Rather, the presentation is confined to the sampling and analytical results and will describe only those aspects of the project which are necessary to understand the sampling procedures and the analytical results. In the main body of the report (Section 6), the analytical results are tabularized in concentration units such as mg/m3 (milligrams per cubic meter) and ug/g (micro grams per gram) to facilitate interpretation and comparisons. A much more complete presentation of the data is available in the supplemental data appendix (e.g., filter weights, gas flow volumes, raw instrumental data).

Before work began on the site, Radian prepared a Quality Assurance Program Plan (hereinafter referred to as the QAPP).[1] It was reviewed and approved by Enviresponse and EPA. In addition to giving specific details of the quality assurance/quality control structure, it also presented:

- An overview of the incineration process;

- A description of all sampling equipment and procedures;

- A description of all analytical procedures;

- Protocols for all sampling handling and storage;

- Reporting requirements including data quality evaluation protocols; and

- Staffing, schedule and safety programs.

Since the program was carried out essentially as specified in the QAPP, most of that information will not be repeated here; the reader is referred to that document for detailed information. Any deviations from the QAPP will be duly noted. Also, Shirco is submitting a separate report to Enviresponse giving details about the incinerator and how it was operated.

The overall objective of the sampling and analytical task was to characterize the performance of the Shirco incinerator with respect to process gases, particulate matter, volatile and semi-volatile toxic organics, metals (with special emphasis on lead), PCBs (poly chlorinated biphenyls) and dioxins and furans. Upwind and downwind (of the site) sampling was also conducted for particulate matter, metals and PCBs to determine overall site emissions. During the first week, November 3-6, 1987, the incinerator was operated (by Shirco) in a steady state while repeated (triplicate) sampling was conducted by Radian; this testing established the reproducibility and a range of variation of the results for the incinerator and sampling methods. During the second week of sampling, November 9-13, 1987, the incinerator was parametrically run over a range of operating conditions (e.g., fuel rates, temperatures, residence times) while Radian carried out a somewhat less comprehensive testing program.

Essentially all of the original objectives set out in the QAPP were met. Conclusions are presented in Section 2, tabularized data are given in Section 6, and a discussion of the results is presented in Section 7. The quality of the data is assessed in Section 8.

2.0 SUMMARY OF RESULTS AND CONCLUSIONS

A voluminous quantity of sampling and analytical data were generated in this study. While certain aspects of the performance of the Shirco Infrared Incinerator Process are in question or debatable, some definitive statements can be made.

- Particulate emission from the off-gas stack were all below 0.03 gr/dscf, well below the RCRA limit of 0.08 gr/dscf. During the second week, all concentrations were below 0.01 gr/dscf.

- Most of the metals contained in the feed material remained in the furnace ash. Lead on the flue-gas particulate averaged about 300 ug/m3 which translated into about 60 mg/hr. The feed rate was about 70 lbs/hr (32 kg/hr).

- Very low levels of any pollutants were found in scrubber liquors.

- PCBs in the feed varied from 10 to 669 ppm. Greater than 99.99% destruction was achieved during the SITE testing. When the primary chamber was operated at 900°F (the lowest temperature condition), 0.1-3 ppm levels of PCBs were detected in the ash.

- Essentially no dioxins or furans were detected during this study.

- Only very small amounts of semi-volatile organics (mostly phthalates) were detected in the off-gases.

- As expected, a variety of volatile organic substances were detected in the primary off-gases. The major components, sometimes at levels above 100 ug/m3, were halomethanes (e.g., chloromethane) chlorinated hydrocarbons (e.g., chlorobenzene), certain hydrocarbons (e.g., toluene) and some partially oxygenated hydrocarbons (e.g., acrolein).

- Many volatile organic compounds were substantially reduced through the secondary combustion chamber (e.g., toluene was reduced from 13 ug/m3 to below 1 ug/m3). However, certain species appeared to be unaffected or increase. The impact of secondary combustion should be considered on a compound-by-compound basis.

- Leaching studies indicated that, on one occasion, lead slightly exceeded the RCRA regulatory limit of 5 mg/L; more often the lead concentration was about 20% of the limit. Most metals, including lead and chromium, were found at higher concentrations in the TCLP leachate than in the EP toxicity leachate.

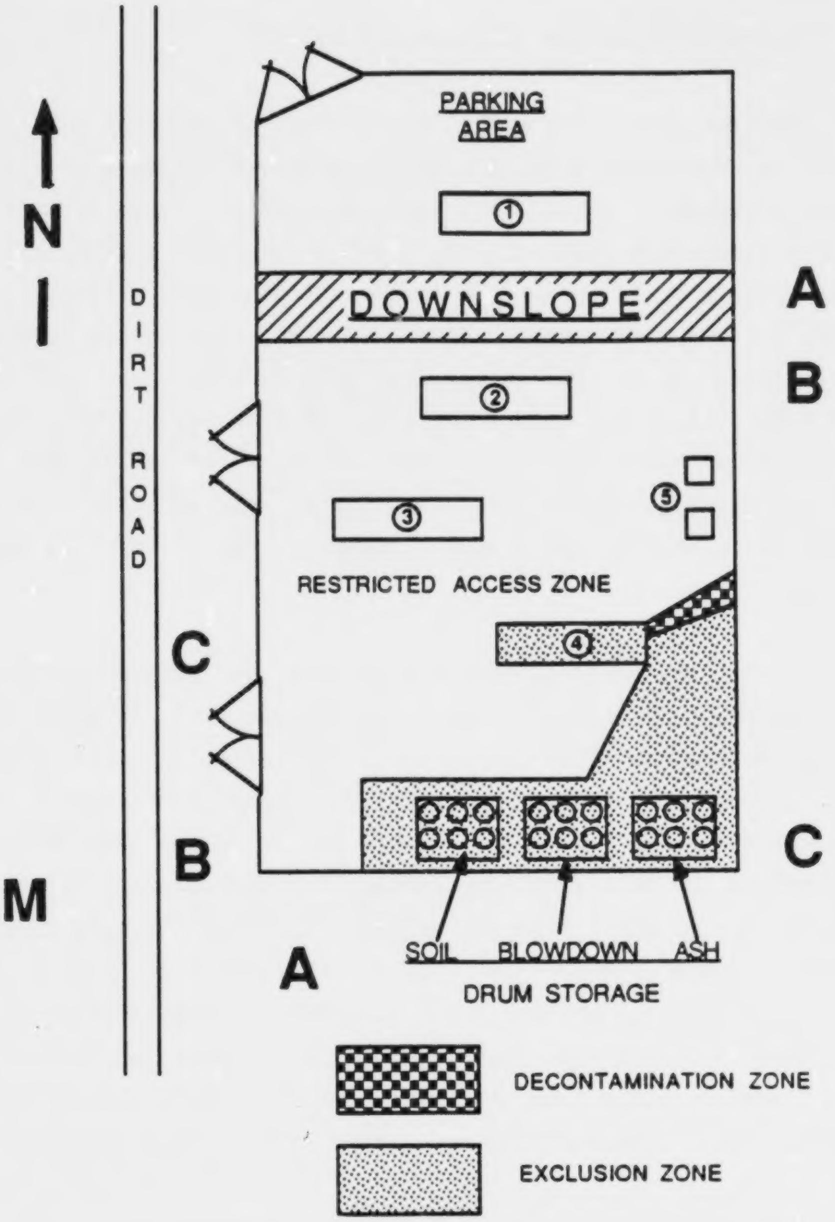
- NO_x emission levels were at about 120 ppm and 80 ppm when the secondary combustion chamber was operated at 2200°F and 1800°F, respectively.

3.0 DESCRIPTION OF THE INCINERATION PROCESS

Several weeks before any incineration was to take place, contaminated soil was excavated from representative areas at the Rose Township-Demode Road site, screened to exclude any particles (rocks) larger than one inch, and loaded into fifty-five gallon lined, steel drums. The drums were transported about two hundred yards and stored in an exclusion zone. About one week before the testing was to begin, Shirco located their mobile incinerator immediately adjacent to the contaminated soil storage area. The exclusion zone was defined by Enviresponse included the contaminated soil storage area, the feed material handling area and the inside of the incinerator trailer. Only Shirco personnel handled the contaminated soil and Radian personnel were not permitted in the exclusion zone. Figure 3-1 is a schematic representation of the incineration site.

During the testing, the feed material was transferred from the drums into five gallon buckets and, after weighing, fed onto the metal furnace belt. Feed rates ranged from 62.95 lbs/hr to 128.57 lbs/hr.[2] The contaminated soil then moved on the belt into the primary chamber, where it was heated with infrared radiatsion. In all tests except No. 19 on the last day, the depth of the soil bed on the belt was 1-1/2 inches; for test No. 19, it was set at 1 inch. Both the residence time and temperature in the primary chamber were varied over a range of conditions. Residence times were usually 20 minutes, but some tests were run at 10, 15 and 25 minutes. Temperatures in the primary chamber ranged from 900°F to 1600°F. Table 3-1, which reports many of these data, was taken directly from the Shirco report to Enviresponse and lists the test number, date, time and test conditions of each test.[2]

The pyrolized soil left the primary chamber, still on the furnace belt, and emptied into a lined, steel drum; see Figure 3-2 for a schematic representation of the incinerator. The off-gases from the primary chamber passed through a duct and into a secondary combustion chamber where propane was added and the mixture burned at either 2200 or 1800°F; see Table 3-1.


N
PARKING AREA
①
DOWNSLOPE
A
B
②
DIRT ROAD
⑤
③
RESTRICTED ACCESS ZONE
C
④
B
C
M
A
SOIL
BLOWDOWN
ASH
DRUM STORAGE
DECONTAMINATION ZONE
EXCLUSION ZONE

(NOTE: A, B, C refer to upwind/downwind positions of hivol samples on different days. M. Indicates location of meteorological station.)

Figure 3-1. Schematic of Rose Township Site

TABLE 3-1. INCINERATOR LOGS (From Shirco Report[2])

OPERATION SUMMARY
SHIRCO PORTABLE DEMONSTRATION UNIT
ROSE TOWNSHIP, MI

DATE	TIME PERIOD (HRS)	EMISSIONS TEST NO.	PRIMARY CHAMBER RES. TIME (MIN.)	PRIMARY CHAMBER TEMPERATURE (F)	FEED RATE (LBS/HR)	SECONDARY CHAMBER TEMPERATURE (F)	SECONDARY CHAMBER RES. TIME (SEC.)
11-03-87	10:20-11:40	-	20	1600	88.89	2200	
11-03-87	11:40-18:25	1	20	1600	66.53	2200	2.60
11-04-87	07:33-09:45	-	20	1600	69.09	2200	
	09:45-20:13	2	20	1600	73.11	2200	
11-05-87	07:55-08:16	-	20	1600	82.86	2200	
	08:16-18:37	3	20	1600	77.41	2200	
11-06-87	09:55-10:32	-	20	1600	128.57	2200	
	10:32-15:31	3	20	1600	70.23	2200	
11-09-87	09:34-10:35	10	20	1200	78.68	1800	
	11:26-12:27	14	15	1200	95.41	1800	
	13:45-14:47	5	20	1400	65.81	2200	
11-10-87	09:30-10:30	6	15	1600	110.77	1800	
	11:20-12:20	8	15	1600	120.00	1800	
	13:13-14:14	15	15	1200	100.33	1800	
	15:07-16:07	18	20	900	80.00	1800	
11-11-87	09:04-10:05	7	10	1600	114.10	2200	
	11:30-12:31	9	25	1200	62.95	2200	
	13:20-14:20	11	20	1200	73.85	2200	
	15:10-16:10	13	15	1200	88.29	2200	
11-12-87	10:27-11:27	16	25	900	65.00	2200	
	12:35-13:35	17	20	900	70.00	1800	
11-13-87	10:12-11:12	19	15	1600	88.29	1800	

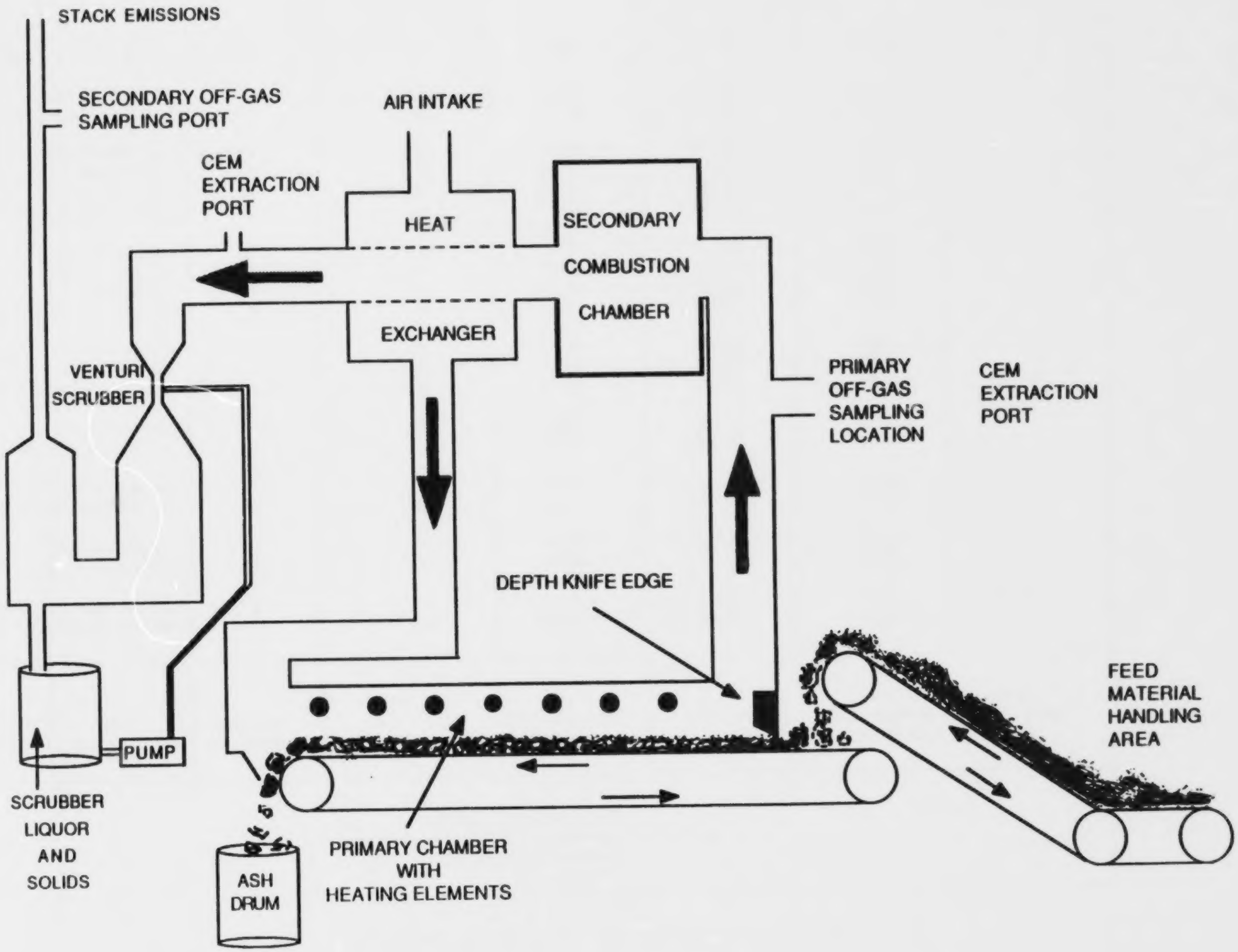

STACK EMISSIONS
SECONDARY OFF-GAS SAMPLING PORT
CEM EXTRACTION PORT
AIR INTAKE
HEAT
EXCHANGER
SECONDARY
COMBUSTION
CHAMBER
VENTURI SCRUBBER
PRIMARY OFF-GAS SAMPLING LOCATION
CEM EXTRACTION PORT
DEPTH KNIFE EDGE
FEED MATERIAL HANDLING AREA
PUMP
SCRUBBER LIQUOR AND SOLIDS
ASH DRUM
PRIMARY CHAMBER WITH HEATING ELEMENTS

Figure 3-2. Schematic of Shirco Incinerator Showing Sampling Locations

Residence time in the secondary chamber averaged 2.6 seconds.[2] The combustion gases then passed through a wet venturi scrubber where they were cooled and most of the particulate matter removed. The saturated (with water) gases were finally vented to the atmosphere through a four inch pipe (the stack). The scrubber liquor was recycled through a holding tank where the solids settled out. At the end of each run, the ash and scrubber effluent (containing very small amounts of solids) were transferred to drums and stored in a separate portion of the exclusion zone.

4.0 DESCRIPTION OF THE SAMPLING PROGRAM

4.1 Sampling Locations and Frequency

The primary objective of Radian's effort at Rose Township was to characterize the efficiency of the incinerator and to quantify the emissions from the process. Radian also placed upwind and downwind high volume (hivol) samplers to measure anthropogenic particulate matter and PCBs generated at the site during the incineration activities. A detailed description of each of the sampling procedures is given in the QAPP; the discussion in this section is intended only to provide a general overview of the basic sampling methodology for someone who has not read the QAPP. In addition, any deviation from the procedures set out in the QAPP are mentioned.

A summary of the streams sampled and the species or properties being determined in each of the streams is given in Table 4-1. The sampling points are identified in Figure 3-1.

In order to determine the fate of the components of the feed material (the contaminated soil), a representative sample was taken every thirty minutes and composited; at the end of the run, the mixture was homogenized, and a one liter portion preserved for analysis. The sample was analyzed for metals, PCBs, dioxins, volatiles and other components of interest to this study; see Table 4-1. As mentioned in Section 3, the total mass flow of feed material during each run was also measured; see Table 3-1. Therefore, the mass flow in the system may be related back to the feed rate.

At the end of each run, composite samples of the two other solid streams, furnace ash and scrubber solids, were also collected and were scheduled for analyses similar to those for the feed material; see Table 4-1. The quantity of scrubber solids available after each run (generally less than fifty grams) precluded gathering enough samples to accommodate all of the scheduled analyses. Since the scrubber water would have leached out most of

TABLE 4-1. SUMMARY OF SAMPLING PLAN

Sampled Stream	Feed Material	Furance Ash	Primary Off-Gas	Secondary Off-Gas	Scrubber Make-Up	Scrubber Liquor	Scrubber Solids	Ambient Air
Mass Flow	A		S	A				A
Metals	A	A	S	S	A	A	S	A
Volatiles	A	A	S	S	A	A		
Semi-Volatiles	A	A	S	S	A	A		
PCB's	A	A	S	S	A	A		A
PDD/PCDF	A	A	S	S	A	A		
TCLP-Metals	A	A				A		
TCLP-Volatiles	A	A				A		
EP-Metals	A	A				A		
TSS					A	A		
TDS					A	A		
TOC					A	A		
Ultimate	A							
Ash	A	A						
Density	A	A						
Chlorine	A	A	S	S	A	A		
Chromium			S	S				
pH					A	A		
Moisture	A	A						

S - Sampled during site testing only (11/4 - 11/6).
A - sampled during all of testing.
TSS - Total suspended solids.
TDS - Total dissolved solids.
TOC - Total organic carbon
Ultimate includes C,H,O,N,S, Ash, Moisture and higher heating value (HHV).

the soluble species, metals analyses was given top priority and consumed all of the available sample.

In order to characterize the performance of the scrubber, samples of the scrubber make-up water and water from the holding tank (scrubber liquor) were taken at the end of each run. These waters were analyzed for volatiles, semi-volaties, PCBs, dioxins, metals and other components of interest; see Table 4-1.

Samples of the process gas were taken at two different points; see Figure 3-1. The first sample was drawn from the duct leading from the primary chamber to the secondary combustion chamber. The gases at this point were hot, usually 1600°F, and heavily ladened with particulate material. Components of interest at this point included the particulate matter itself and the associated metals, volatiles, semi-volatiles, PCBs, dioxins and furans, HCl, Cr(+6), and a special sampling for vapor phase lead (Pb). Identical and simultaneous sampling (except for the lead sampling) was carried out on the secondary combustion chamber off-gases (i.e,, the stack gases). During the first week when the SITE testing was being conducted to determine performance characteristics of the incinerator, all of the process gas testing described above was conducted in triplicate. During the second week, when different operational conditions of the incinerator were being tested, the process gas testing was curtailed to only particulate matter sampling of the primary and secondary gases.

During both phases of the testing, additional samples of the primary and secondary off-gases were continuously withdrawn by a separate system and analyzed by continuous emission monitors (CEMs) for oxygen, carbon monoxide, carbon dioxide, nitrogen oxides and total hydrocarbons. These data were collected as five minute averages. Also, upwind and downwind hivol samplers were set up just beyond the perimeters of the incineration area; they tested the ambient air for total particulate matter (including metals) and PCBs. They were allowed to run all day unless the wind direction changed (as monitored by a meteorological station established at the site) by more than 90 degrees.

4.2 Sampling Methodology

Particulate material was collected in the process gases using EPA Reference Method 5. In this method, a small portion of the gas/particle mixture was withdrawn isokinetically from the gas stream, conducted down an inert probe and filtered at 120°C (248°F). The filter was subsequently removed, dried and weighed. The probe and nozzle were brushed and washed with acetone; the catch was taken to dryness and the weight of residue was added to the weight gain of the filter to determine the total weight of particulate material sampled. In the sampling train, after the gases had been filtered, they passed through chilled water impingers and a desiccant to remove the water vapor. The dry gas volume was then determined with a dry gas meter, while the weight gain of the impingers and desiccant was used to determine the amount of moisture in the original gas. Particle loading results have been reported in conventional mass concentration units of grains per dry standard cubic feet (gr/dscf).

Volatile organics in the flue gases were sampled using the VOST (volatile organic sampling train) method. In this method, a small sample of the gas to be sampled was drawn at a rate of one liter per minute through a glass wool plug to remove the particulate material and conducted down a glass probe. Volatile organics were removed from the gas in sorbent resin traps maintained at 20°C. The first trap contained Tenax resin and the second trap contained Tenax followed by petroleum-based charcoal. Some organics may also be sorbed into the water condensate which forms in the chilled resin traps; this condensate is collected separately. A normal run consisted of several sets of VOST tubes (usually six) each sampling about 20 liters of dry gas as measured with a dry gas meter after the water has been removed (condensed out). When sampling had been completed, the resin traps and combined condensate were sealed individually and returned immediately to the laboratory for analysis.

Semi-volatile organics, dioxins and furans (PCDD/PCDF), and PCBs were collected from the process gases using a modified EPA Method 5 (MM5) methodology. The apparatus was similar to that used for the particulate material collection described above (EPA Method 5) with the addition of a water cooled condenser and a chilled XAD-2 resin trap immediately following the heated filter. The filter was maintained at 205°C (400°F). To improve detection limits, large volumes (at least ten cubic meters) of gas were sampled using the source assessment sampling system; the combination will be referred to as MM5-SASS. All semi-volatile organics (generally defined as all organic species with boiling points above 100°C) were either condensed with the water and/or sorbed onto the resin. The condensate liquor and XAD-2 resin were later co-extracted to isolate the organic fraction.

Soluble chromium (Cr+6) was determined with another modification of the EPA Method 5 described above (MM5-Cr). In this case, the particulate filter was removed and the sampled gas and particle steam impinged directly into a basic (NaOH) solution which quantitatively removed all of the chromium. The entire sampling train, including the nozzle, was made of glass to prevent contamination from any type of steel. Soon after the sampling was completed, the impinger and probe and nozzle wash fractions were combined, and the chromium was fixed by chelating it with ammonium pyrrolidine dithiocarbamate; the complex was then immediately extracted into methyl isobutyl ketone where it was stable.

Solids (feed material, ash, scrubber solids) and liquids (scrubber make-up and holding tank water) were sampled with a scoop, trier or dipper. Representative samples of composited material were taken and placed in specially prepared amber glass bottles.

Process gases were analyzed for certain components (see Section 4-1) by extracting samples of the gases and delivering them via heat traced teflon tubing to a bank of instruments. Before analysis, the sampled gas was conditioned by removing the water. Portions of the clean, dry gas were then sent to each of the five continuous emission monitors (CEMs) for analysis. The data from the instruments were recorded both on strip chart recorders and in a data logging computer. Elaborate calibration and drift check procedures were carried out each day to ensure that the instruments were operating properly and accurately.

Ambient air sampling (for particulate material, metals and PCBs) was carried out using high volume (hivol) samplers. They were located upwind and downwind from the incineration site and moved daily (if necessary) as determined by meteorological data and predictions of wind directions. On any day, if there was a major shift in wind direction (>90 degrees), the samplers were shut off. They collected sample each day the incinerator was operating. One upwind/downwind pair collected ambient particulate matter on glass fiber filters at a rate of about 50 scfm; this material was subsequently weighed and analyzed for metals. Another pair of upwind/downwind hivol samplers was modified and contained a sandwich of polyurethane foam (PUF)/Florisil organic sorbent/PUF to quantitatively capture PCBs. The flow rate was adjusted to 8 scfm. After sampling, the PUF plugs and florisil for each sampler were placed together in a specially cleaned glass bottle and returned to the laboratory to be co-extracted for PCBs.

4.3 Sample Handling

All liquid and solid samples collected on site were placed in prelabled containers (e.g., specially washed amber glass bottles, void-of-air vials). Each container was assigned a unique identifying code, and the sample number, type of sample and weight, along with the time and the initials of the person who collected it, were logged into a master sample log book. Before storage or shipment, each container was sealed (with tape) and that seal was secured with a chain of custody band.

Samples which had to be extracted or analyzed within a short period of time (a few days to less than a month) were packed in coolers (with ice to maintain 40°F) and shipped by overnight express to the laboratory where the work was to be performed. All other samples (none of which required cooling) were placed in labeled boxes and retained by the sampling crew for return with them to Radian's Austin facility at the end of the sampling period.

5.0 ANALYTICAL PROCEDURES

The exact analytical protocols for each type of sample were set out in the QAPP. Essentially all extraction and analytical methods referred to published standard operating procedures (SOPs). Only a very brief review of the preparation and analysis given each type of sample will be given in this report. Many of the methods used in this study are summarized in the publication "Test Methods for Evaluating Solid Waste," U.S. EPA, OSWER, SW-846, 3rd Edition November 1986 (referred to as SW-846).[3]

The weight of particulate material collected in the EPA Method 5 train was determined by desiccating and weighing the tarred filter to a constant weight. The impinger solutions were evaporated and the residue desiccated and weighted to constant weight. In other solutions (scrubber liquors), the total suspended solids (TSS) were determined by filtration with subsequent drying and weighing. Total dissolved solids (TDS) were determined, after filtration, by taking the solution to dryness and weighing the residue to constant weight. pH was determined with a pH meter and chloride was measured by ion chromatography.

There were two types of volatile samples. One type were those collected from gas streams by VOST sampling. The VOST tubes were thermally desorbed (SW-846, Method 5040), concentrated with purge traps and analyzed by GC/MS (SW-846, Method 8240). The second type were the liquid and solid samples which contained light organic species. For these samples, the volatiles were quantitatively removed from the sample by an inert gas purge-and-trap procedure (SW-846, Method 8240) and also analyzed by GC/MS. The results have been reported as the RCRA Appendix IX list of volatile compounds which were listed in the QAPP (Referred to there as the priority pollutants plus the next ten most prominent species, V-PP+10).

The semi-volatile organic compounds (roughly defined as those species with boiling points above 100°C) may occur in either solids (e.g., waste feed, ash, scrubber solids), in liquids (e.g., scrubber water, impinger solutions), or trapped on resins (XAD-2). They were extracted from solid and resin samples with organic solvents by Soxhlet extraction (SW-846, Method 3540). The aqueous samples were extracted with a continuous liquid-liquid procedure (SW-846 Method 3520). The same extraction procedures were also used for dioxins and furans (PCDD/PCDFs), and for some samples which were to be analyzed for both semi-volatiles and PCDD/PCDFs, (the extract was split in half for the separate analyses). Once in the organic solvent, the samples were concentrated and analyzed for the semi-volatile species by GC/MS (SW-846, Method 8270). The results have been reported as the RCRA Appendix IX list of semi-volatile compounds which were listed in the QAPP (Referred to there as the priority pollutants plus the next ten most prominent species, V-PP+10).

Before analysis, the PCDD/PCDF fraction of the extract was further treated by a column clean-up procedure (SW-846, Method 8280). This method used a matrix-specific extraction and an analyte-specific clean-up column. Analysis was by high resolution capillary column gas chromatography/low resolution mass spectroscopy (HRGC/LRMS), and the results reported for the principal tetra-, penta-, hexa-, and octa-chlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs).

PCBs (polychlorinated biphenyls) in the solid waste feed, flue gas, furnace ash, scrubber water, scrubber make-up water and the ambient air PUF plugs were analyzed following EPA Method 680. Solid samples and PUF plugs were first Soxhlet extracted (SW-846, Method 3540), and the aqueous solutions were liquid/liquid extracted (SW-846, Method 3520). Extracts from different components of the flue gas sampling system for any one run were combined prior to analysis. Because of interfering species present in these types of samples, all analyses were carried out using HRGC/LRMS with the mass spectrometer operating in the specific ion monitoring (SIM) mode.

Metals analyses were carried out on flue gas particulate matter, ambient air particulate matter, feed material, ash, and scrubber make-up water, holding tank water and the scrubber solids. The solid samples and the filters with the particulate matter on them were digested with strong acids (SW-846, Methods 3050 and 3020). The resulting liquids were then analyzed using inductively coupled (argon) plasma emission spectroscopy (ICPES) (SW-846, Method 6010). With this technology, a small sample of the liquid was injected into the flame of a plasma torch and the emitted light simultaneously spectroscopically analyzed for multiple metals. To achieve greater accuracy and lower detection limits, several metals were analyzed using a graphite furnace (arsenic, antimony, lead, selenium; SW-846, Methods 7060, 7041, 7421, 7740, respectively), and mercury was determined using the cold vapor method (SW-846, Method 7470/7471).

Soluble chromium (Cr^{+6}), on the flue gas particulate matter, required special analytical techniques. Since the Cr^{+6} tends to degrade with time, as soon as possible after sampling the chromium must be fixed; see Section 4. It was then analyzed using a flame atomic absorption spectroscopy specially modified for chromium analysis.

The solid feed material, furnace ash and scrubber water were analyzed according to the RCRA Characteristic method for EP Toxicity (SW-846, Method 1310). In this procedure, the soluble toxic metals are extracted with acetic acid, and the metals content of the extract is determined using either ICEPES, cold vapor (for Hg) or a graphite furnace as described above. The metals of concern in this study were As, Ba, Cd, Cr, Pb, Hg, Se and Ag.

A second extraction was also carried out on the same four samples mentioned in the preceding paragraph. The RCRA Toxic Characteristics Leaching Procedure (TCLP) (40 CFR 261, Appendix) is a more complex acetic acidic buffered, void of air extraction and is used for the same eight metals above and all of the RCRA Appendix IX volatiles and semi-volatiles. The metals were analyzed as described above for EP Toxicity, while the volatiles and semi-volatile organic species were analyzed according to the protocols previously referred to (SW-846, Methods 8240 and 8270, respectively).

Total chlorine was determined in the feed material, ash and scrubber solids using the combustion/absorption/precipitation methodology (ASTM D808-81). The precipitation used silver chloride, and if the the chloride concentration was too low, ion chromatography was substituted (ASTM D4327-84).

The total ash content of the feed material and furnace ash and scrubber solids was determined using method ASTM D3174. In this procedure, the sample was ignited and ashed at 775°C in a muffle furnace. The residue was then weighed.

The feed material and furnace ash were also subjected to ultimate analysis for C, H, O, S, N, Ash and Moisture. The methods used are a conglomerate of several ASTM methods. The higher heating value (HHV) for the feed material was also determined using ASTM Method D2015-77. A bomb calorimeter was used in this procedure.

Finally, the bulk density of the feed material and furnace ash was determined using standard laboratory procedures for the gravimetric/volumetric method. A 250 ml sample was used.

6.0 SUMMARY OF RESULTS

In this section, the measured and calculated results are presented for all of the sampling and analyses connected with the Rose Township-Demode Rode site. Brief descriptions of the sampling and analytical methods have been given in earlier sections, and detailed protocols were given in the QAPP. Here, the information will be confined to results presented in the units which are most convenient for understanding the incineration process; in general, data will be given in metric concentration units.

Where appropriate and available, average blank values have been subtracted from the original primary data, so that the values appearing in the tables represent the best numbers obtainable from these data. Original numbers direct from the laboratory or field measurements (e.g., mass of analyte, sample volume) are available in the supplemental data Appendix. Observations about relationships, trends, mass flows and anomolies are discussed in Section 7. A discussion of the extensive quality assurance/quality control (QA/QC) effort is given in Section 8.

The data have been grouped by the type of analysis (e.g., metals, PCBs); sub-groups have been distinguished according to the type of sample (e.g., furnace ash, scrubber water). Table 6-1 is intended to serve as a roadmap to the many tables in this Section.

6.1 Particulate Loading and HCl in the Flue Gases

Table 6-2 presents the mass particulate loadings in the primary and secondary off-gas streams determined by EPA Method 5. Both the original particle loadings and those corrected to 7% oxygen have been included. The flue gas flow rates, which are useful for mass flow calculations have been included. Also, on the same table, the HCl concentration in the off-gases are reported. Ambient particle loadings have been included with the metals analysis; see Table 6-6.

TABLE 6-1. GUIDE TO OTHER TABLES IN SECTION 6

Table Numbers	Type of Analysis or Result	Sub-Sections
6-2	Particulate Loading & HCl	Flue Gas
6-3 -> 6-9	Metals (not special Pb, Cr)	
6-3		Feed Material
6-4		Flue Gas Particulate
6-5		Furnace Ash
6-6		Ambient Air
6-7		Scrubber Make-Up
6-8		Scrubber Liquors
6-9		Scrubber Solids
6-10 -> 6-15	PCBs	
6-10		Feed Material
6-11		Flue Gas
6-12		Scrubber Make-Up
6-13		Scrubber Liquors
6-14		Furnace Ash
6-15		Ambient air
6-16 -> 6-20	Dioxins and Furans	
6-16		Feed Material
6-17		Flue Gas
6-18		Scrubber Make-Up
6-19		Scrubber Liquors
6-20		Furnace Ash
6-21 -> 6-26	Semi-Volatile Organic Pollutants	
6-21		Feed Material
6-22		Flue Gas
6-23		Scrubber Make-Up
6-24		Scrubber Liquors
6-25		Furnace Ash

(Continued)

TABLE 6-1. (Continued)

Table Numbers	Type of Analysis or Result	Sub-Sections
6-26 -> 6-30	Volatile Organic Pollutants	
6-26		Feed Material
6-27		Flue Gas
6-28		Scrubber Make-Up
6-29		Scrubber Liquors
6-30		Furnace Ash
6-31 -> 6-36	Leachability Determinations	
6-31	-- EP Toxicity	Feed Material
6-32	-- EP Toxicity	Scrubber Liquors
6-33	-- EP Toxicity	Furnace Ash
6-34	-- TCLP	Feed Material
6-35	-- TCLP	Scrubber Liquors
6-36	-- TCLP	Furnace Ash
6-37 -> 6-38	Solids Characterizations	
6-37		Feed Material
6-38		Furnace Ash
6-39 -> 6-40	Liquids Characterizations	
6-39		Scrubber Make-Up
6-40		Scrubber Liquors
6-41	Soluble Chromium Determination	
6-42	Vapor Phase Lead Determination	
6-43	Continuous Flue Gas Monitor Results	
6-44	Meteorological Hivol Data & Location	

TABLE 6-2. CONCENTRATION OF PARTICULATE MATERIAL AND HCl IN FLUE GAS

					Particulate(+)			HCl	
Location	Date	Time	Flue Gas Flowrate	Oxygen Content	Measured Concentration	7% O2 Concentration	Measured Emission Rate	Concentration	Emission Rate
			(dscfm)	(%)	(gr/dscf)	(gr/dscf)	(Lb/Hr)	(ppmv)	(Lb/Hr)
Primary	11/3/87	1723-1823	36.2	17.6	0.072	0.30	0.022	183	0.038
Primary	11/5/87	1300-1400	37.2	17.0	0.32	1.1	0.10	472	0.10
Primary	11/6/87	1105-1306	36.8	17.6	0.35	1.4	0.11	642	0.14
Stack	11/3/87	1630-1750	139	8.5	0.0093	0.010	0.011	0.4	0.0004
Stack	11/5/87	1301-1354	119	9.8	0.024	0.030	0.024	2	0.0013
Stack	11/6/87	1057-1255	132	7.2	0.015	0.015	0.017	3	0.0022
Stack	11/9/87	0936-1036	131	10.7	0.0061	0.0083	0.0068	---	---
Stack	11/9/87	1126-1226	122	12.3	0.0042	0.0068	0.0044	---	---
Stack	11/9/87	1346-1446	120	7.5	0.0048	0.0050	0.0049	---	---
Stack	11/10/87	0931-1031	127	8.6	0.0045	0.0051	0.0049	---	---
Stack	11/10/87	1121-1221	119	8.3	0.0029	0.0032	0.0030	---	---
Stack	11/10/87	1313-1413	99	9.8	0.0035	0.0044	0.0030	---	---
Stack	11/10/87	1508-1608	136	11.1	0.0028	0.0040	0.0032	---	---
Stack	11/11/87	0904-1004	140	8.9	0.0073	0.0084	0.0088	---	---
Stack	11/11/87	1132-1232	129	9.8	0.0082	0.0100	0.0091	---	---
Stack	11/11/87	1322-1422	100	9.2	0.0078	0.0093	0.0067	---	---
Stack	11/11/87	1511-1611	109	9.0	0.0078	0.0091	0.0073	---	---
Stack	11/12/87	1028-1128	125	9.0	0.0071	0.0083	0.0077	---	---
Stack	11/12/87	1235-1335	109	11.4	0.0055	0.0080	0.0052	---	---
Stack	11/13/87	1010-1110	112	9.3	0.0055	0.0066	0.0053	---	---

(+) Although most of the results in this report are given in metric units. English units are so common for these quantities that they have been retained. For conversion to metric units, multiply values in (gr/dscf) by 2288 to obtain value in (mg/m3); multiply (Lb/Hr) by 0.453 to obtain value in (Kg/Hr).

6.2 Concentrations of Metals (Except Special Pb and Cr Determinations)

Table 6-3 presents the results of the ICPES metals analysis of the feed material in units of ug/g (or ppm, by weight). Each separate analysis, representing a composite sample from a different day or different run, defines a column in the table.

Table 6-4 presents the results of the ICPES metals analysis of the particulate material and probe and nozzle washes collected on the EPA Method 5 sampling trains. The results are given in units of ug/m3 for each element. Since the filter was maintained at about 120°C (248°F), these data report the metal concentration in the flue gas at such temperatures (i.e., metallic vapors at 120°C would not be included). Each separate analysis, representing a different Method 5 run, defines a column in the table.

Table 6-5 presents the results of the ICPES metals analysis of the furnace ash in units of ug/g (or ppm, by weight). Each separate analysis, representing a composite sample from a different day or different run, defines a column in the table.

Table 6-6 presents the results of the total mass loading and metals analysis of the material on the upwind and downwind ambient air filters. Data are in units of ug/m^3. Each separate day defines a column in the table.

Table 6-7 and 6-8 present the results of the ICPES metals analysis of the scrubber make-up water and scrubber liquors, respectively. Data are given in units of mg/L. For the scrubber make-up water, drawn from the same source throughout the program, samples were only taken once per day, and each day defines a column in Table 6-7. For the scrubber liquors, each separate analysis, representing a different run, defines a column in Table 6-8.

Table 6-9 presents the results of the ICPES metals analysis on the scrubber solids. Data are given in units of mg/g. Only small amounts of

TABLE 6-3. RESULTS OF THE METALS ANALYSIS ON THE FEED MATERIAL

Analyte	Detection Limit+ (ug/g)	Date: Time:	11/3/87 2030 (ug/g)	11/4/87 2030 (ug/g)	11/5/87 2000 (ug/g)	11/6/87 1830 (ug/g)
Antimony	0.30		26	1.7	1.3[a]	1.1[a]
Arsenic	2.0		12	11	11	9.8[a]
Barium	0.87		700	600	500	940
Beryllium	0.097		ND	ND	ND	ND
Cadmium	0.29		8.6	2.6	3.4	11
Chromium	0.87		82	72	80	180
Copper	0.97		15	15	16	28
Lead	2.0		3000*	1400	550	1100
Mercury	0.050		0.080[a]	0.10[a]	0.12[a]	0.13[a]
Nickel	1.9		21	21	24	34
Selenium	0.3		ND	ND	ND	ND
Silver	0.87		2.2[a]	2.1[a]	ND	1.9[a]
Thallium	8.7		ND	ND	ND	ND
Vanadium	1.9		16	18	26	20
Zinc	0.58		280	290	240	320

(Continued)

*Sample reanalyzed; reported value confirmed.

+Method (not instrumental) detection limit varied for each sample because of dilution factors; average value used in this column.

TABLE 6-3. (Continued)

Analyte	Detection Limit+ (ug/g)	Date: Time:	11/9/87 1110 (ug/g)	11/9/87 1245 (ug/g)	11/9/87 1700 (ug/g)	11/10/87 1045 (ug/g)
Antimony	0.30		1.7	2.2	1.6[b]	1.4[a]
Arsenic	2.0		10	12	11	10
Barium	0.87		700	510	550	420
Beryllium	0.097		ND	ND	ND	ND
Cadmium	0.29		3.9	3.3	4.8	3.2
Chromium	0.87		80	100	71	66
Copper	0.97		15	21	16	16
Lead	2.0		640	870	740	620
Mercury	0.050		0.070[a]	0.30	0.090[a]	0.080
Nickel	1.9		25	32	24	22
Selenium	0.3		ND	ND	ND	ND
Silver	0.87		1.6[a]	2.2[a]	2.5[a]	2.2[a]
Thallium	8.7		ND	ND	ND	ND
Vanadium	1.9		21	21	21	22
Zinc	0.58		290	310	290	250

Analyte	Detection Limit + (ug/g)	Date: Time:	11/10/87 1245 (ug/g)	11/10/87 1450 (ug/g)	11/10/87 1645 (ug/g)	11/11/87 1030 (ug/g)
Antimony	0.30		1.4[a]	0.80[a]	2.1	1.2[a]
Arsenic	2.0		10	9.3[a]	11	8.2[a]
Barium	0.87		570	510	660	590
Beryllium	0.097		ND	ND	ND	ND
Cadmium	0.29		4.1	4.0	3.2	3.5
Chromium	0.87		77	79	82	81
Copper	0.97		16	15	15	16
Lead	2.0		500	370	590	620
Mercury	0.050		ND	0.080[a]	0.12[a]	0.080[a]
Nickel	1.9		21	32	23	23
Selenium	0.3		ND	ND	ND	ND
Silver	0.87		1.8[a]	4.4	2.0[a]	2.0[a]
Thallium	8.7		ND	ND	ND	ND
Vanadium	1.9		22	19	21	21
Zinc	0.58		280	310	310	350

(Continued)

+Method (not instrumental) detection limit varied for each sample because of dilution factors; average value used in this column.

TABLE 6-3. (Continued)

Analyte	Detection Limit[+] (ug/g)	Date: Time:	11/11/87 1245 (ug/g)	11/11/87 1450 (ug/g)	11/11/87 1645 (ug/g)	11/12/87 1145 (ug/g)
Antimony	0.30		0.70[a]	ND	1.5	1.2[a]
Arsenic	2.0		13	11	11	9.9[a]
Barium	0.87		720	420	790	410
Beryllium	0.097		ND	ND	ND	ND
Cadmium	0.29		5.0	420	3.4	2.6
Chromium	0.87		120	66	74	59
Copper	0.97		22	16	15	18
Lead	2.0		590	610	470	290
Mercury	0.050		0.14[a]	0.080[a]	ND	0.12[a]
Nickel	1.9		110	23	19	21
Selenium	0.3		ND	ND	ND	ND
Silver	0.87		1.5[a]	2.4[a]	1.3[a]	1.2[a]
Thallium	8.7		ND	ND	ND	ND
Vanadium	1.9		22	20	20	21
Zinc	0.58		590	240	200	220

Analyte	Detection Limit[+] (ug/g)	Date: Time:	11/12/87 1345 (ug/g)	11/13/87 1230 (ug/g)
Antimony	0.30		1.3[a]	0.60[a]
Arsenic	2.0		10	11
Barium	0.87		650	390
Beryllium	0.097		ND	ND
Cadmium	0.29		4.0	4.3
Chromium	0.87		74	85
Copper	0.97		17	34
Lead	2.0		660	390
Mercury	0.050		0.11[a]	0.11[a]
Nickel	1.9		22	57
Selenium	0.3		ND	ND
Silver	0.87		3.4[a]	6.2
Thallium	8.7		ND	ND
Vanadium	1.9		19	17
Zinc	0.58		400	240

[a] Indicates a value less than 5 times the detection limit.

[b] Indicates that the spike recovery was not within acceptable limits.

ND - not detected.

+Method (not instrumental) detection limit varied for each sample because of dilution factors; average value used in this column.

TABLE 6-4. RESULTS OF METALS ANALYSIS ON THE EPA METHOD 5 FILTERS

Location:	Primary	Primary	Primary	Secondary	Secondary	Secondary
Date:	11/3/87	11/5/87	11/6/87	11/3/87	11/5/87	11/6/87
Time:	1723-1823	1300-1400	1105-1306	1630-1750	1301-1354	1057-1255
Total Mass (g):	0.0889	0.1530	0.2017	0.0174	0.0186	0.0134
Volume (m^3):	0.5388	0.6236	0.6412	1.1215	0.6431	0.7513
Analyte	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)
Antimony	52	120	120	7.6	20	21
Arsenic	11	27	28	0.98	1.9	1.7
Barium	1100	2200	2000	7.0	22	17
Beryllium	<0.37	<0.32	<0.33	<0.18	<0.31	<0.27
Cadmium	39	160	190	8.3	37	32
Chromium	72	150	130	45	560 a	77
Copper	17	35	34	6.9	56 a	10
Lead	650	1500	1900	380	260	250
Mercury	0.26	0.35	0.64	0.16	0.031	<0.027
Nickel	41	51	67	12	420 a	6.0
Selenium	11	18	19	3.8	11	7.3
Silver	<3.3	3.2	20	<1.6	<2.8	<2.4
Thallium	<33	11	<30	<16	<28	<24
Vanadium	9.1	11	13	7.1	17	<5.5
Zinc	250	450	440	31	280 a	59

a: Value outside reasonable range and considered in error.

TABLE 6-5. RESULTS OF THE METALS ANALYSIS ON THE FURNACE ASH

Analyte	Detection Limit+ (ug/g)	Date: Time:	11/3/87 2030 (ug/g)	11/4/87 2030 (ug/g)	11/5/87 2000 (ug/g)	11/6/87 1830 (ug/g)
Antimony	0.30		ND	ND	ND[b]	ND
Arsenic	2.0		21	15[a]	14	9.3
Barium	0.87		1400	1200	1000	1100
Beryllium	0.097		ND	ND	ND	ND
Cadmium	0.29		5.6	5.5	6.7	4.4
Chromium	0.87		78	79	110	71
Copper	0.97		14	16	14	19
Lead	2.0		1100	1300	1100	420
Mercury	0.050		ND	ND	ND	ND
Nickel	1.9		46	39	30	31
Selenium	0.3		ND	ND[b]	ND	ND
Silver	0.87		3.1[a]	3.1[a]	5.8	3.6[a]
Thallium	8.7		ND	ND	ND	ND
Vanadium	1.9		26	34	30	31
Zinc	0.58		360	410	340	350

(Continued)

+Method (not instrumental) detection limit varied for each sample because of dilution factors; average value used in this column.

TABLE 6-5. (Continued)

Analyte	Detection Limit+ (ug/g)	Date: Time:	11/9/87 1100 (ug/g)	11/9/87 1245 (ug/g)	11/9/87 1700 (ug/g)	11/10/87 1045 (ug/g)
Antimony	0.30		ND	ND	ND	ND
Arsenic	2.0		12	12	14	21
Barium	0.87		970	1000	950	790
Beryllium	0.097		ND	ND	ND	ND
Cadmium	0.29		6.7	7.5	7.3	3.8
Chromium	0.87		77	100	81	62
Copper	0.97		15	17	16	12
Lead	2.0		1100	1000	1600	840
Mercury	0.050		ND	ND	ND	ND
Nickel	1.9		51	87	35	29
Selenium	0.3		ND	ND	ND	ND
Silver	0.87		2.6[a]	3.1[a]	3.3[a]	3.0[a]
Thallium	8.7		ND	ND	ND	ND
Vanadium	1.9		23	26	23	33
Zinc	0.58		380	420	420	380

Analyte	Detection Limit+ (ug/g)	Date: Time:	11/10/87 1245 (ug/g)	11/10/87 1450 (ug/g)	11/10/87 1645 (ug/g)	11/11/87 1030 (ug/g)
Antimony	0.30		ND	ND	ND	ND
Arsenic	2.0		23	18	11	20
Barium	0.87		1100	1800	720	940
Beryllium	0.097		ND	ND	ND	ND
Cadmium	0.29		4.2	6.7	5.5	5.2
Chromium	0.87		68	84	86	75
Copper	0.97		13	17	18	15
Lead	2.0		800	1000	1000	1700
Mercury	0.050		ND	ND	ND	ND
Nickel	1.9		26	31	25	35
Selenium	0.3		ND	ND	ND	ND
Silver	0.87		4.7	3.7[a]	3.3[a]	3.3[a]
Thallium	8.7		ND	ND	ND	ND
Vanadium	1.9		31	33	18	30
Zinc	0.58		420	640	380	400

(Continued)

+Method (not instrumental) detection limit varied for each sample because of dilution factors; average value used in this column.

TABLE 6-5. (Continued)

Analyte	Detection Limit+ (ug/g)	Date: Time:	11/11/87 1245 (ug/g)	11/11/87 1450 (ug/g)	11/11/87 1645 (ug/g)	11/12/87 1145 (ug/g)
Antimony	0.30		ND	ND	ND	1.6
Arsenic	2.0		17	16	14	11
Barium	0.87		1000	760	690	940
Beryllium	0.097		ND	ND	ND	ND
Cadmium	0.29		7.6	5.5	6.5	6.3
Chromium	0.87		78	76	74	85
Copper	0.97		15	15	17	19
Lead	2.0		1200	1200	2000	860
Mercury	0.050		ND	ND	ND	ND
Nickel	1.9		61	28	49	49
Selenium	0.3		ND	2.2	ND	ND
Silver	0.87		3.7[a]	3.5[a]	3.1[a]	4.2
Thallium	8.7		ND	ND	ND	ND
Vanadium	1.9		26	24	24	22
Zinc	0.58		360	510	350	410

Analyte	Detection Limit+ (ug/g)	Date: Time:	11/12/87 1345 (ug/g)	11/13/87 1230 (ug/g)
Antimony	0.30		2.1	3.2
Arsenic	2.0		10	19
Barium	0.87		940	1800
Beryllium	0.097		ND	ND
Cadmium	0.29		5.4	3.7
Chromium	0.87		94	79
Copper	0.97		19	17
Lead	2.0		1400	1500
Mercury	0.050		ND	ND
Nickel	1.9		33	36
Selenium	0.3		ND	ND
Silver	0.87		3.3[a]	2.4[a]
Thallium	8.7		ND	10[a]
Vanadium	1.9		18	28
Zinc	0.58		410	440

[a] Indicates a value less than 5 times the detection limit.

[b] Indicates that the spike recovery was not within acceptable limits.

ND - not detected.

+Method (not instrumental) detection limit varied for each sample because of dilution factors; average value used in this column.

TABLE 6-6. RESULTS OF METALS ANALYSIS ON AMBIENT PARTICULATE

Date:	11/3/87		11/4/87		11/5/87		11/6/87	
Location:	Downwind	Upwind	Downwind	Upwind	Downwind	Upwind	Downwind	Upwind
Total mass (g)	0.0529	0.0399	0.0360	0.0295	0.0143	0.0097	0.0134	0.0053
Volume (m^3)	440	561	547	757	681	788	254	433
Analyte	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)
Antimony	<0.0091	<0.0071	<0.0073	<0.0053	<0.0059	<0.0051	<0.014	<0.0083
Arsenic	<0.0045	<0.0036	<0.0036	<0.0026	<0.0029	<0.0025	<0.0094	<0.0065
Barium	<0.025	0.028	0.024	<0.015	<0.016	<0.014	<0.043	<0.025
Beryllium	0.0023	0.0018	<0.0022	<0.0016	<0.0018	<0.0015	<0.0047	<0.0028
Cadmium	<0.0082	0.023	<0.0066	<0.0048	<0.0053	<0.046	<0.014	<0.0083
Chromium	<0.025	<0.020	0.029	<0.015	0.034	<0.014	<0.043	0.051
Copper	0.36	0.13	0.091	0.10	0.070	0.055	0.063	0.083
Lead	0.10	0.039	0.062	0.032	0.013	0.013	0.094	0.025
Mercury	0.00086	0.00028	0.0018	0.0011	0.0011	0.00065	<0.00047	0.0011
Nickel	0.068	<0.043	<0.044	0.033	<0.035	<0.030	<0.094	<0.065
Selenium	<0.0091	<0.0071	<0.0073	<0.0053	<0.0059	<0.0051	0.014	<0.0083
Silver	<0.025	<0.020	<0.020	<0.015	<0.016	<0.014	0.13	0.037
Thallium	<0.25	<0.20	<0.20	<0.15	<0.16	<0.14	<0.43	<0.25
Vanadium	<0.054	<0.043	<0.044	0.033	<0.035	<0.030	<0.094	<0.065
Zinc	0.16	0.098	0.097	0.073	0.035	0.028	0.12	0.039

Date:	11/9/87		11/10/87		11/11/87		11/12/87		11/13/87	
Location:	Downwind	Upwind	Downwind	Upwind	Downwind	Upwind	Downwind	Upwind	Downwind	Upwind
Total mass (g)	0.0208	0.0024	0.0160	0.0039	0.0095	0.0042	0.0143	0.0110	0.0265	0.0138
Volume (m^3)	216	317	492	292	339	209	139	266	141	272
Analyte	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)	(ug/m^3)
Antimony	<0.017	<0.011	<0.0073	<0.012	<0.011	<0.017	<0.026	<0.014	<0.025	<0.013
Arsenic	<0.011	<0.0075	<0.0049	<0.0082	<0.0071	<0.011	<0.017	<0.0090	<0.0169	<0.0088
Barium	<0.051	<0.035	<0.022	<0.038	<0.032	<0.053	<0.079	<0.041	0.20	<0.040
Beryllium	<0.0055	<0.0038	<0.0024	<0.0041	<0.0035	<0.0057	<0.0086	<0.0045	<0.0085	<0.0044
Cadmium	<0.017	0.038	<0.0073	<0.012	<0.011	<0.017	<0.026	<0.014	<0.025	<0.013
Chromium	<0.051	<0.035	<0.022	<0.038	<0.032	<0.053	<0.079	<0.041	<0.078	<0.040
Copper	<0.055	<0.038	<0.024	0.044	<0.035	<0.057	<0.086	<0.045	0.13	0.092
Lead	0.034	0.060	0.039	0.041	0.032	0.045	0.11	0.053	0.27	0.073
Mercury	<0.00055	<0.00038	<0.00024	<0.00041	<0.00035	<0.00057	<0.00086	<0.00045	<0.00085	0.00044
Nickel	<0.11	<0.075	<0.049	<0.082	<0.071	<0.11	<0.17	<0.090	<0.17	<0.088
Selenium	<0.017	0.022	<0.0089	0.016	0.018	0.036	0.055	0.029	0.054	0.032
Silver	<0.051	<0.035	<0.022	<0.038	<0.032	<0.053	<0.079	<0.041	<0.078	<0.040
Thallium	0.51	<0.35	<0.22	<0.38	<0.32	<0.53	<0.79	<0.41	<0.78	<0.40
Vanadium	<0.11	<0.075	<0.049	<0.082	<0.071	<0.11	<0.17	<0.090	<0.17	<0.088
Zinc	0.041	0.057	0.030	<0.025	<0.021	<0.034	0.072	0.045	0.57	0.11

TABLE 6-7. RESULTS OF THE METALS ANALYSIS ON THE SCRUBBER MAKEUP

Analyte	Detection Limit (mg/L)	Date: Time:	11/3/87 2030 (mg/L)	11/4/87 2030 (mg/L)	11/5/87 2000 (mg/L)	11/6/87 1830 (mg/L)
Antimony	0.0030		ND	ND	ND	ND
Arsenic	0.0020		0.010	0.0080[a]	0.0090[a]	0.010
Barium	0.0090		0.15	0.14	0.13	0.14
Beryllium	0.0010		ND	ND	ND	0.0030[a]
Cadmium	0.0030		0.0040[a]	ND	ND	0.010[a]
Chromium	0.0090		0.015[a]	0.025[a]	0.027[a]	ND
Copper	0.010		ND	ND	ND	0.0060[a]
Lead	0.0020		0.0030[a]	ND	ND	ND
Mercury	0.00020		ND	ND	ND	0.020
Nickel	0.020		0.030[a]	ND	ND	ND
Selenium	0.0030		ND	ND	ND	0.023[a]
Silver	0.0090		0.047	0.026[a]	0.024[a]	ND
Thallium	0.090		ND	ND	ND	0.040[a]
Vanadium	0.020		ND	ND	0.050[a]	0.043
Zinc	0.0060		0.038	0.039	0.019[a]	

(Continued)

TABLE 6-7. (Continued)

Analyte	Detection Limit (mg/L)	Date: Time:	11/9/87 1045 (mg/L)	11/10/87 1445 (mg/L)	11/11/87 1015 (mg/L)	11/12/87 1150 (mg/L)
Antimony	0.0030		ND[b]	ND	ND	ND
Arsenic	0.0020		0.0090[a]	0.010	0.0090[a]	0.0080[a]
Barium	0.0090		0.13	0.14	0.13	0.14
Beryllium	0.0010		ND	ND	ND	ND
Cadmium	0.0030		0.0030[a]	ND	0.0030[a]	0.0050[a]
Chromium	0.0090		0.034[a]	0.022[a]	0.040[a]	ND
Copper	0.010		0.010[a]	0.020[a]	0.010[a]	0.010[a]
Lead	0.0020		0.075	ND	ND	0.0040[a]
Mercury	0.00020		ND	ND	ND	ND
Nickel	0.020		ND	ND	ND	ND
Selenium	0.0030		ND	ND	ND	ND
Silver	0.0090		0.026[a]	0.023[a]	0.025[a]	0.051
Thallium	0.090		ND	ND	ND	ND
Vanadium	0.020		0.020[a]	0.020[a]	ND	0.020[a]
Zinc	0.0060		0.032	0.023[a]	0.019[a]	0.027[a]

Analyte	Detection Limit (mg/L)	Date: Time:	11/13/87 1130 (mg/L)
Antimony	0.0030		ND
Arsenic	0.0020		0.0090[a]
Barium	0.0090		0.14
Beryllium	0.0010		ND
Cadmium	0.0030		ND
Chromium	0.0090		ND
Copper	0.010		0.010[a]
Lead	0.0020		0.011
Mercury	0.00020		ND
Nickel	0.020		ND
Selenium	0.0030		ND
Silver	0.0090		0.018[a]
Thallium	0.090		ND
Vanadium	0.020		0.020[a]
Zinc	0.0060		0.029[a]

ND - not detected.

[a] Indicates a value less than 5 times the detection limit.

[b] Indicates that the spike recovery was not within acceptable limits.

TABLE 6-8. RESULTS OF THE METALS ANALYSIS ON THE SCRUBBER LIQUOR

Analyte	Detection Limit (mg/L)	Date: Time:	11/3/87 2030 (mg/L)	11/4/87 2030 (mg/L)	11/5/87 2000 (mg/L)	11/6/87 1830 (mg/L)
Antimony	0.0030		0.14	0.20	0.19	0.38
Arsenic	0.0020		0.064	0.061	0.050	0.082
Barium	0.0090		1.4	1.0	0.96	2.2
Beryllium	0.0010		ND	ND	ND	ND
Cadmium	0.0030		0.044	0.080	0.072	0.087
Chromium	0.0090		0.47	0.33	0.38	0.79
Copper	0.010		0.12	0.080	0.050	0.17
Lead	0.0020		1.5	1.2	1.0	1.8
Mercury	0.00020		ND	ND	ND	0.0016
Nickel	0.020		0.30	0.25	0.14	0.41
Selenium	0.0030		0.019	0.023	0.040	0.022
Silver	0.0090		0.010[a]	0.032[a]	0.020[a]	0.031[a]
Thallium	0.090		ND	ND	ND	ND
Vanadium	0.020		0.090[a]	0.11	0.090[a]	0.12
Zinc	0.0060		0.56	0.45	0.33	0.86

(Continued)

TABLE 6-8. (Continued)

Analyte	Detection Limit (mg/L)	Date: Time:	11/9/87 1110 (mg/L)	11/9/87 1245 (mg/L)	11/9/87 1700 (mg/L)	11/10/87 1045 (mg/L)
Antimony	0.0030		0.062	0.032	0.096	0.044
Arsenic	0.0020		0.024	0.018	0.025	0.026
Barium	0.0090		0.55	0.62	0.65	0.87
Beryllium	0.0010		ND	ND	ND	ND
Cadmium	0.0030		0.015[a]	0.016	0.013[a]	0.023
Chromium	0.0090		0.13	0.082	0.19	0.15
Copper	0.010		0.040[a]	0.030[a]	0.030[a]	0.050
Lead	0.0020		0.37	0.28	0.48	0.52
Mercury	0.00020		0.00080[a]	0.00080[a]	0.0016	0.0010
Nickel	0.020		0.090[a]	0.070[a]	0.060[a]	0.10
Selenium	0.0030		ND	ND	ND	0.0080[a]
Silver	0.0090		0.016[a]	0.052	0.019[a]	0.021[a]
Thallium	0.090		ND	ND	ND	ND
Vanadium	0.020		0.030[a]	0.030[a]	0.080[a]	0.030[a]
Zinc	0.0060		0.19	0.15	0.18	0.27

Analyte	Detection Limit (mg/L)	Date: Time:	11/10/87 1245 (mg/L)	11/10/87 1450 (mg/L)	11/10/87 1645 (mg/L)	11/11/87 1030 (mg/L)
Antimony	0.0030		0.045	0.051	0.063	0.13
Arsenic	0.0020		0.021	0.023	0.025	0.027
Barium	0.0090		0.79	0.86	0.84	0.47
Beryllium	0.0010		ND	ND	ND	ND
Cadmium	0.0030		0.032	0.079	0.021	0.030
Chromium	0.0090		0.13	0.14	0.16	0.21
Copper	0.010		0.040[a]	0.050	0.060	0.030[a]
Lead	0.0020		0.40	0.68	0.58	0.49
Mercury	0.00020		0.0013	0.0011	0.0011	0.00080[a]
Nickel	0.020		0.090[a]	0.14	0.090[a]	0.050[a]
Selenium	0.0030		0.018	0.0070[a]	ND	0.0030[a]
Silver	0.0090		0.023[a]	0.042[a]	0.022[a]	0.021[a]
Thallium	0.090		ND	ND	ND	ND
Vanadium	0.020		0.030[a]	0.040[a]	0.030[a]	0.030[a]
Zinc	0.0060		0.21	0.32	0.21	0.12

(Continued)

TABLE 6-8. (Continued)

Analyte	Detection Limit (mg/L)	Date: Time:	11/11/87 1245 (mg/L)	11/11/87 1450 (mg/L)	11/11/87 1645 (mg/L)	11/12/87 1145 (mg/L)
Antimony	0.0030		0.084	0.082	0.096	0.078
Arsenic	0.0020		0.023	0.020	0.018	0.015
Barium	0.0090		0.56	0.45	0.53	0.42
Beryllium	0.0010		ND	ND	ND	ND
Cadmium	0.0030		0.034	0.023	0.029	0.0050[a]
Chromium	0.0090		0.19	0.14	0.12	0.12
Copper	0.010		0.030[a]	0.030[a]	0.030[a]	0.030[a]
Lead	0.0020		0.51	0.39	0.37	0.34
Mercury	0.00020		0.0011	0.00090[a]	0.0012	0.00040[a]
Nickel	0.020		0.060[a]	0.050[a]	0.050[a]	0.060[a]
Selenium	0.0030		0.0030[a]	ND	ND	ND
Silver	0.0090		0.021[a]	0.023[a]	0.024[a]	0.061[a]
Thallium	0.090		ND	ND	ND	ND
Vanadium	0.020		0.040[a]	0.040[a]	0.040[a]	0.040[a]
Zinc	0.0060		0.15	0.14	0.13	0.11

Analyte	Detection Limit (mg/L)	Date: Time:	11/12/87 1345 (mg/L)	11/13/87 1230 (mg/L)
Antimony	0.0030		0.034	0.047
Arsenic	0.0020		0.013	0.011
Barium	0.0090		0.48	0.20
Beryllium	0.0010		ND	ND
Cadmium	0.0030		0.0040[a]	0.010[a]
Chromium	0.0090		0.087	0.057
Copper	0.010		0.030[a]	0.020[a]
Lead	0.0020		0.19	0.12
Mercury	0.00020		0.00060[a]	0.00040[a]
Nickel	0.020		0.050[a]	0.040[a]
Selenium	0.0030		ND	0.010[a]
Silver	0.0090		0.023[a]	0.017[a]
Thallium	0.090		ND	ND
Vanadium	0.020		0.030[a]	0.040[a]
Zinc	0.0060		0.11	0.056

ND - not detected.

[a] Indicates a value less than 5 times the detection limit.

TABLE 6-9. RESULTS OF METALS ANALYSIS ON THE SCRUBBER SOLIDS

Analyte	Detection Limit (ug/g)	Date: Time:	11/3/87 2030 (ug/g)	11/4/87 2030 (ug/g)
Antimony	6.0+		13	14[a]
Arsenic	0.30		43	34
Barium	0.90		910	1600
Beryllium	0.10		ND	ND
Cadmium	0.30		31	37
Chromium	0.90		460	230
Copper	1.0		170	110
Lead	5.0		1200	870
Mercury	0.050		0.88	0.65
Nickel	2.0		310	240
Selenium	0.30		ND[a]	ND
Silver	0.90		7.8	6.5
Thallium	9.0		ND	ND
Vanadium	2.0		34	31
Zinc	0.60		770	620

[a] Indicates that the spike recovery was not within acceptable limits.
ND - not detected.

NOTE: Not enough scrubber solids to perform analysis 11/5 through 11/13.
+ Higher than usual DL due to nature and quantity of sample.

scrubber solids were recoverable from the "bottom of the barrel" on the first two days (<10g). Even less was found on subsequent days, and even that had the appearance of Fe_2O_3. Further collection was suspended.

6.3 Concentrations of PCBs (Polychlorinated Biphenyls)

A sample containing PCBs is first extracted in an organic solvent. That sample is then concentrated and a reference compound added (spiked) to calibrate matrix effects and instrument response. Also, the GC/MS is separately calibrated to each analyte. Finally, the sensitivity of the detector is adjusted so that it is not saturated by the largest analyte. All of these factors are taken into account by the internal software when determining a "less than" value if the analyte is not detected. Thus, for PCBs, they may vary from run to run and from compound to compound.

The total PCBs in a sample are usually defined as the total equivalent effective toxicity. Since the monochloro- and dichloro-congeners are not nearly so toxic, in summing all the congeners, they are divided by 50 and 5, respectively, before being summed into the total (40 CFR 761.3); less than values are not included in the totals. This effective toxicity total is reported in the PCB tables. This formula (there is no well agreed methodology for adding such columns of PCB data) can produce some odd looking results (e.g., Table 6-11, column 1) and the reader is advised to develop other summing formula if this approach proves misleading.

Table 6-10 presents the results of the PCB analysis (EPA Method 680) of the feed material in units of ng/g (or ppb, by weight). Each separate analysis, representing a composite sample from a different day or different run, defines a column in the table.

Table 6-11 presents the results of the PCB analysis of the particulate material collected by the modified EPA Method 5 source assessment sampling system (MM5-SASS). The extracts from the filter, XAD-2 resin, resin

TABLE 6-10. RESULTS OF PCB ANALYSIS OF FEED MATERIAL

Date:	11/3/87	11/4/87	11/5/87	11/9/87	11/9/87	11/9/87
Time:	2030	2030	2000	1100	1245	1700
ANALYTE	(ng/g)	(ng/g)	(ng/g)	(ng/g)	(ng/g)	(ng/g)
Monochlorobiphenyl	<700	<900	<800	<800	<700	<700
Dichlorobiphenyl	<700	<800	<700	<700 a	<600 a	<600 a
Trichlorobiphenyl	1400	5200	3400	40000	3600	34000
Tetrachlorobiphenyl	8800	30000	17000	200000 a	24000 a	22000 a
Pentachlorobiphenyl	<1000	<2000	<2000	45000 a	<1000 a	<1000 a
Hexachlorobiphenyl	<3000	<3000	<300	12000	<3000	<2000
Heptachlorobiphenyl	<4000	<5000	<4000	<4000	<4000	<3000
Octachlorobiphenyl	<4000	<6000	<5000	<5000	<4000	<4000
Nonachlorobiphenyl	<6000	<8000	<7000	<7000	<6000	<5000
Decachlorobiphenyl	<6000	<7000	<6000	<7000	<6000	<6000
Total	10200	35200	20400	297000	27600	56000

Date:	11/10/87	11/10/87	11/10/87	11/10/87	11/11/87	11/11/87
Time:	1045	1245	1450	1645	1030	1245
ANALYTE	(ng/g)	(ng/g)	(ng/g)	(ng/g)	(ng/g)	(ng/g)
Monochlorobiphenyl	<800	<800	<900	<700	<800	<600
Dichlorobiphenyl	1400	2600	2600	<600	<700	<600
Trichlorobiphenyl	67000	46000	46000	47000	56000	66000
Tetrachlorobiphenyl	300000	210000	210000	220000	260000	300000
Pentachlorobiphenyl	68000	46000	44000	48000	61000	70000
Hexachlorobiphenyl	16000	8000	8200	12000	14000	16000
Heptachlorobiphenyl	<4000	<5000	<5000	<4000	<4000	3800
Octachlorobiphenyl	<5000	<5000	<7000	<4000	<5000	<4000
Nonachlorobiphenyl	<7000	<7000	<8000	<6000	<7000	<6000
Decachlorobiphenyl	<6000	<7000	<8000	<6000	<7000	<5000
Total	451280	310520	308720	327000	391000	455800

Date:	11/11/87	11/11/87	11/12/87	11/12/87	11/13/87
Time:	1450	1700	1145	1345	1230
ANALYTE	(ng/g)	(ng/g)	(ng/g)	(ng/g)	(ng/g)
Monochlorobiphenyl	<4000	<900	<60	<600	<900
Dichlorobiphenyl	<4000	90000	<300 a	<600 a	<800 a
Trichlorobiphenyl	96000	90000	55000	3200	40000
Tetrachlorobiphenyl	480000	390000	230000 a	17000	190000
Pentachlorobiphenyl	93000	85000	54000 a	<1000 a	37000
Hexachlorobiphenyl	<2000	19000	17000	<3000	3800 b
Heptachlorobiphenyl	<2000	<5000	9200	<4000	<5000
Octachlorobiphenyl	<2000	<6000	1700	<4000	<6000
Nonachlorobiphenyl	<4000	<8000	<500	<6000	<8000
Decachlorobiphenyl	<3000	<8000	<500	<6000	<8000
Total	669000	602000	366900	20200	270800

a: Matrix spike recovery outside of recommended limits
b: Value less than 5 times detection limit
LTB: Value measured less than value measured for blank

TABLE 6-11. RESULTS OF THE PCB ANALYSIS OF THE FLUE GAS

	Primary					
Date:	11/3/87		11/4/87		11/5/87	
Time:	1208-1655		1607-1926		0815-1200	
Volume(dscm):	11.659		9.812		11.981	
Flowrate(dscmm):	1.02		1.02		1.10	
	Mass Detected (ng)	Flue Gas Conc. (ng/m3)	Mass Detected (ng)	Flue Gas Conc. (ng/m3)	Mass Detected (ng)	Flue Gas Conc. (ng/m3)
Monochlorobiphenyls	84	7.2	<20	<2.0	<30	<2.5
Dichlorobiphenyls	<20	<1.7	<20	<2.0	<30	<2.5
Trichlorobiphenyls	<30	<2.6	<30	<3.1	<50	<4.2
Tetrachlorobiphenyls	<50	<4.3	<50	<5.1	<80	<6.7
Pentachlorobiphenyls	<30	<2.6	<30	<3.1	<60	<5.0
Hexachlorobiphenyls	<60	<5.1	<60	<6.1	<100	<8.3
Heptachlorobiphenyls	<90	<7.7	<90	<9.2	<200	<17
Octachlorobiphenyls	<100	<8.6	<100	<10	<200	<17
Nonachlorobiphenyls	<100	<8.6	<100	<10	<300	<25
Decachlorobiphenyls	<100	<8.6	<100	<10	<200	<17
Total	1.7	0.14	---	---	---	---

	Secondary					
Date:	11/3/87		11/4/87		11/5/87	
Time:	1228-1800		1608-1924		0938-1330	
Volume(dscm):	7.861		6.928		6.873	
Flowrate(dscmm):	3.60		3.26		3.14	
	Mass Detected (ng)	Flue Gas Conc. (ng/m3)	Mass Detected (ng)	Flue Gas Conc. (ng/m3)	Mass Detected (ng)	Flue Gas Conc. (ng/m3)
Monochlorobiphenyls	<20	<2.5	<20	<2.9	<20	<2.9
Dichlorobiphenyls	<20	<2.5	<20	<2.9	<20	<2.9
Trichlorobiphenyls	<40	<5.1	<40	<5.8	160	23
Tetrachlorobiphenyls	<60	<7.6	<70	<10	310	45
Pentachlorobiphenyls	<50	<6.4	<50	<7.2	<40	<5.8
Hexachlorobiphenyls	<80	<10	<90	<13	<70	<10
Heptachlorobiphenyls	<100	<13	<100	<14	<100	<15
Octachlorobiphenyls	<100	<13	<200	<29	<100	<15
Nonachlorobiphenyls	<200	<25	<200	<29	<200	<29
Decachlorobiphenyls	<200	<25	<200	<29	<200	<29
Total	---	---	---	---	470	68

condensate and the probe, nozzle and glassware rinses were all combined before analysis so that the PCB values reported represent all of the PCBs which passed the nozzle tip. The results are given in units of ng/m^3 in the flue gas. Each separate analysis, representing a different run, defines a column in the table.

Table 6-12 and 6-13 present the results of PCB analysis on the scrubber make-up water and scrubber liquor, respectively. Each separate analysis, representing a different day or different run, defines a column in the table. The results are reported in units of ng/L. In general, there was not enough of the scrubber solids to provide a sample for all types of analyses called for in the QAPP. Metals were given the top priority, and there was never enough sample available for PCB analysis of scrubber solids.

Table 6-14 presents the results of the PCB analysis of the furnace ash in units of ng/g (or ppb, by weight). Each separate analysis, representing a composite sample from different run, defines a column in the table.

Table 6-15 presents the results of the PCB analysis for the ambient air samples. The extracts of the hivol PUF plugs and the Florisil resins were combined before analysis so the values given in the table represent the total of the PCBs that were taken in by the hivol sampler. The results are given in units of ng/m^3.

6.4 Concentrations of Dioxins and Furans (PCDD/PCDFs)

Table 6-16 presents the results of the PCDD/PCDF analyses (SW-846, Method 8280) of the feed material in units of ng/g (or ppb, by weight). Each separate analysis, representing a composite sample from a different day or different run, defines a column in the table.

Table 6-17 presents the results of the PCDD/PCDF analyses of the particulate material collected by the modified EPA Method 5 source assessment

TABLE 6-12. RESULTS OF PCB ANALYSIS OF SCRUBBER MAKE-UP WATER

Date:	11/3/87	11/4/87	11/5/87	11/9/87
Time:	2030	2030	2000	1700
ANALYTE	(ng/L)	(ng/L)	(ng/L)	(ng/L)
Monochlorobiphenyl	<40	<30	---	<40
Dichlorobiphenyl	<30 a	<20 a	---	<40 a
Trichlorobiphenyl	<70	<50	---	<80
Tetrachlorobiphenyl	<100	<80	---	<100
Pentachlorobiphenyl	<70 a	<60 a	---	<80 a
Hexachlorobiphenyl	<200	<100	---	<200
Heptachlorobiphenyl	<200	<100	---	<200
Octachlorobiphenyl	<200	<100	---	<200
Nonachlorobiphenyl	<300	<200	---	<300
Decachlorobiphenyl	<300	<200	---	<300
Total	---	---	---	---

Date:	11/10/87	11/11/87	11/12/87	11/13/87
Time:	1630	1645	1345	1130
ANALYTE	(ng/L)	(ng/L)	(ng/L)	(ng/L)
Monochlorobiphenyl	<30	<8	<60	<6
Dichlorobiphenyl	<20 a	<7 a	<60	<5
Trichlorobiphenyl	<50	<10	<100	<10
Tetrachlorobiphenyl	<80	<20	<200	<20
Pentachlorobiphenyl	<60 a	<20 a	<100	<10
Hexachlorobiphenyl	<100	<30	<200	<20
Heptachlorobiphenyl	<200	<40	<300	<30
Octachlorobiphenyl	<200	<40	<300	<40
Nonachlorobiphenyl	<200	<70	<400	<50
Decachlorobiphenyl	<200	<60	<400	<50
Total	---	---	---	---

a: Matrix spike recovery outside of recommended limits
LTB: Value measured less than value measured for blank
---: sample contaminated or destroyed

TABLE 6-13. RESULTS OF PCB ANALYSIS OF SCRUBBER LIQUORS

Date:	11/3/87	11/4/87	11/5/87	11/9/87	11/9/87	11/9/87
Time:	2030	2030	2000	1045	1100	1700
ANALYTE	(ng/L)	(ng/L)	(ng/L)	(ng/L)	(ng/L)	(ng/L)
Monochlorobiphenyl	<20	<30	<20	<6	<30	<20
Dichlorobiphenyl	<20 a	<20 a	<10 a	<6 a	<20 a	<20 a
Trichlorobiphenyl	<40	<30	LTB	<10	<50	<30
Tetrachlorobiphenyl	<70	<50	LTB	<20	<80	<50
Pentachlorobiphenyl	<50 a	<40 a	<70 a	<10 a	<60 a	<40 a
Hexachlorobiphenyl	<100	<70	<60	<30	<100	<60
Heptachlorobiphenyl	<100	<90	<80	<40	<100	<100
Octachlorobiphenyl	<100	<90	<80	<40	<100	<100
Nonachlorobiphenyl	<200	<100	<100	<60	<200	<200
Decachlorobiphenyl	<200	<200	<100	<60	<200	<200
Total	---	---	---	---	---	---

Date:	11/10/87	11/10/87	11/10/87	11/10/87	11/11/87	11/11/87
Time:	1030	1230	1445	1630	1015	1230
ANALYTE	(ng/L)	(ng/L)	(ng/L)	(ng/L)	(ng/L)	(ng/L)
Monochlorobiphenyl	<10	<60	<10	<10	<9	<30
Dichlorobiphenyl	<10 a	<60 a	<30 a	<10 a	<9 a	<20 a
Trichlorobiphenyl	<20	<60	<50	<20	<20	<50
Tetrachlorobiphenyl	<40	<200	<80	<30	<20	<70
Pentachlorobiphenyl	<30 a	<100 a	<60 a	<20 a	<20 a	<50 a
Hexachlorobiphenyl	<50	<200	<100	<40	<30	<100
Heptachlorobiphenyl	<80	<300	<200	<70	<50	<100
Octachlorobiphenyl	<90	<300	<100	<80	<50	<200
Nonachlorobiphenyl	<100	<200	<200	<100	<80	<200
Decachlorobiphenyl	<100	<400	<200	<100	<70	<200
Total	---	---	---	---	---	---

Date:	11/11/87	11/11/87	11/12/87	11/12/87	11/13/87
Time:	1445	1645	1145	1345	1130
ANALYTE	(ng/L)	(ng/L)	(ng/L)	(ng/L)	(ng/L)
Monochlorobiphenyl	<50	<50	<20	<20	<9
Dichlorobiphenyl	<40 a	<40 a	<20 a	<20 a	<8 a
Trichlorobiphenyl	<80	<90	<40	<30	<20
Tetrachlorobiphenyl	<100	<100	<70	<50	<20
Pentachlorobiphenyl	<100 a	<100 a	<50 a	<40 a	<20 a
Hexachlorobiphenyl	<200	<200	<100	<70	<30
Heptachlorobiphenyl	<200	<200	<100	<100	<50
Octachlorobiphenyl	<200	<200	<200	<100	<50
Nonachlorobiphenyl	<300	<300	<200	<200	<80
Decachlorobiphenyl	<300	<300	<200	<100	<70
Total	---	---	---	---	---

a: Matrix spike recovery outside of recommended limits
LTB: Value measured less than value measured for blank

TABLE 6-14. RESULTS OF PCB ANALYSIS OF FURNACE ASH

Date: Time: ANALYTE	11/3/87 2030 (ng/g)	11/4/87 2030 (ng/g)	11/5/87 2000 (ng/g)	11/9/87 1100 (ng/g)	11/9/87 1245 (ng/g)	11/9/87 1700 (ng/g)
Monochlorobiphenyl	<1	<3	<1	<3	<3	<2
Dichlorobiphenyl	<1	<2	<1	<3 a	<3 a	<2 a
Trichlorobiphenyl	7.8 b	17 b	3	<5	20	<4
Tetrachlorobiphenyl	29 b	95 b	<4	<8 a	57 a	<6 a
Pentachlorobiphenyl	<3	<5	<3	<6 a	<6 a	<5 a
Hexachlorobiphenyl	<6	<10	<6	<10	<1	<8
Heptachlorobiphenyl	<8	<10	<8	<20	<20	<10
Octachlorobiphenyl	<8	<10	<7	<20	<20	<10
Nonachlorobiphenyl	<10	<20	<10	<20	<30	<20
Decachlorobiphenyl	<10	<20	<10	<20	<30	<20
Total	36.8	112	3	---	77	---

Date: Time: ANALYTE	11/10/87 1045 (ng/g)	11/10/87 1245 (ng/g)	11/10/87 1450 (ng/g)	11/10/87 1645 (ng/g)	11/11/87 1030 (ng/g)	11/11/87 1245 (ng/g)
Monochlorobiphenyl	<3	<2	<2	6.8 b	<1	<3
Dichlorobiphenyl	<3 a	<2 a	<2 a	21 ab	<1 a	<3 a
Trichlorobiphenyl	<6	<3	<4	240	<2	<6
Tetrachlorobiphenyl	<9 a	<5 a	<6 a	1100 a	<4 a	<9 a
Pentachlorobiphenyl	<6 a	<4 a	<4 a	400 a	<3 a	<7 a
Hexachlorobiphenyl	<10	<7	<8	180	<5	<12
Heptachlorobiphenyl	<20	<9	<10	110	<6	<15
Octachlorobiphenyl	<20	<9	<10	33 b	<6	<14
Nonachlorobiphenyl	<20	<10	<10	6 b	<9	<20
Decachlorobiphenyl	<20	<10	<10	5.5 b	<8	<19
Total	---	---	---	2078.8	---	---

Date: Time: ANALYTE	11/11/87 1450 (ng/g)	11/11/87 1700 (ng/g)	11/12/87 1145 (ng/g)	11/12/87 1345 (ng/g)	11/13/87 1230 (ng/g)
Monochlorobiphenyl	<2	<0.5	1 a	9.4 b	<0.5
Dichlorobiphenyl	<2 a	<0.5 a	3 ab	32 a	<0.4 a
Trichlorobiphenyl	<4	<1 b	38	610	1 b
Tetrachlorobiphenyl	<6 a	<5 b	110 a	2000 a	3.2 ab
Pentachlorobiphenyl	<4 a	<1 a	19 a	500 a	<1 a
Hexachlorobiphenyl	<8	<2	<2	160	<2
Heptachlorobiphenyl	<10	<3	<2	98	<3
Octachlorobiphenyl	<10	<3	<3	21	<3
Nonachlorobiphenyl	<10	<5	<4	<8	<4
Decachlorobiphenyl	<10	<4	<3	<8	<4
Total	---	---	167.7	3395.9	4.2

a: Matrix spike recovery outside of recommended limits
b: Value less than 5 times detection limit

TABLE 6-15. RESULTS OF THE PCB ANALYSIS OF THE AMBIENT AIR

Date:	11/3/87				11/4/87			
Location:	Upwind		Downwind		Upwind		Downwind	
Volume(dscm):	82.55		81.39		103.76		104.02	
Parameter	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)
Monochlorobiphenyls	<20	<0.24	110	1.4	<7.0	<0.067	<10	<0.096
Dichlorobiphenyls	34	0.41	<20	<0.25	<7.0	<0.067	<10	<0.096
Trichlorobiphenyls	400	4.8	110	1.4	88	0.85	78	0.75
Tetrachlorobiphenyls	860	10	76	0.93	280	2.7	260	2.5
Pentachlorobiphenyls	<40	<0.48	<50	0.61	<20	<0.19	<30	<0.29
Hexachlorobiphenyls	<90	<1.1	<97	<1.2	<30	<0.29	<60	<0.58
Heptachlorobiphenyls	<100	<1.2	<100	<1.2	<40	<0.39	<80	<0.77
Octachlorobiphenyls	<100	<1.2	<100	<1.2	<40	<0.39	<70	<0.67
Nonachlorobiphenyls	<100	<1.2	<200	<2.4	<50	<0.48	<100	<0.96
Decachlorobiphenyls	<100	<1.2	<200	<2.4	<50	<0.48	<100	<0.96
Total	1300	15	190	2.3	370	3.5	340	3.2

Date:	11/5/87				11/6/87			
Location:	Upwind		Downwind		Upwind		Downwind	
Volume(dscm):	86.29		99.23		54.74		55.11	
Parameter	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)
Monochlorobiphenyls	<30	<0.35	22	0.22	110	2.0	<20	<0.36
Dichlorobiphenyls	<30	<0.35	<20	<0.20	<10	<0.18	<10	<0.18
Trichlorobiphenyls	<60	<0.70	<30	<0.30	240	4.4	98	1.8
Tetrachlorobiphenyls	<100	<1.2	<50	<0.50	610	11	460	8.3
Pentachlorobiphenyls	<70	<0.81	<40	<0.40	<30	<0.55	<30	<0.54
Hexachlorobiphenyls	<100	<1.2	<70	<0.71	<50	<0.91	<60	<1.1
Heptachlorobiphenyls	<200	<2.4	<100	<1.0	<70	<1.3	<80	<1.5
Octachlorobiphenyls	<200	<2.4	<90	<0.91	<70	<1.3	<80	<1.5
Nonachlorobiphenyls	<200	<2.4	<100	<1.0	<100	<1.8	<100	<1.8
Decachlorobiphenyls	<200	<2.4	<100	<1.0	<100	<1.8	<100	<1.8
Total	<1100	<13	0.44	0.0044	850	16	560	10

(Continued)

TABLE 6-15. (Continued)

Date:	11/9/87				11/10/87			
Location:	Upwind		Downwind		Upwind		Downwind	
Volume(dscm):	39.17		38.97		63.27		61.40	
Parameter	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)
Monochlorobiphenyls	<20	<0.51	470	12	<20	<0.32	48	0.78
Dichlorobiphenyls	<20	<0.51	<20	<0.51	<20	<0.32	76	1.2
Trichlorobiphenyls	110	2.8	140	3.6	120	1.9	290	4.7
Tetrachlorobiphenyls	490	13	710	18	410	6.5	2200	36
Pentachlorobiphenyls	<40	<1.0	<50	<1.3	<40	<0.64	480	7.8
Hexachlorobiphenyls	<70	<1.8	<90	<2.3	<70	<1.1	<80	<1.3
Heptachlorobiphenyls	<90	<2.3	<100	<2.6	<90	<1.4	<100	<1.6
Octachlorobiphenyls	<90	<2.3	<100	<2.6	<90	<1.4	<100	<1.6
Nonachlorobiphenyls	<100	<2.6	<200	<5.2	<100	<1.6	<100	<1.6
Decachlorobiphenyls	<100	<2.6	<100	<2.6	<100	<1.6	<100	<1.6
Total	600	15	860	22	530	8.4	3000	49

Date:	11/11/87				11/12/87			
Location:	Upwind		Downwind		Upwind		Downwind	
Volume(dscm):	45.57		43.36		36.11		35.60	
Parameter	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)
Monochlorobiphenyls	150	3.3	<40	<0.92	<50	<1.4	740	21
Dichlorobiphenyls	<40	<0.88	<30	<0.69	<50	<1.4	<50	<1.4
Trichlorobiphenyls	<70	<1.5	<70	<1.6	<80	<2.2	<80	<2.2
Tetrachlorobiphenyls	240	5.3	470	11	<100	<2.8	<100	<2.8
Pentachlorobiphenyls	<80	<1.8	78	1.8	<100	<2.8	<90	<2.5
Hexachlorobiphenyls	<200	<4.4	<100	<2.3	<200	<5.5	<200	<5.6
Heptachlorobiphenyls	<200	<4.4	<200	<4.6	<300	<8.3	<300	<8.4
Octachlorobiphenyls	<200	<4.4	<200	<4.6	<300	<8.3	<300	<8.4
Nonachlorobiphenyls	<300	<6.6	<300	<6.9	<400	<11	<400	<11
Decachlorobiphenyls	<300	<6.6	<200	<4.6	<300	<8.3	<300	<8.4
Total	240	5.3	550	13	---	---	15	0.42

(Continued)

TABLE 6-15. (Continued)

Date:	11/13/87			
Location:	Upwind		Downwind	
Volume(dscm):	36.93		38.23	
Parameter	Mass Detected (ng)	Ambient Conc. (ng/m3)	Mass Detected (ng)	Ambient Conc. (ng/m3)
Monochlorobiphenyls	<60	<1.6	78	2.0
Dichlorobiphenyls	<50	<1.4	<50	<1.3
Trichlorobiphenyls	<80	<2.2	<100	<2.6
Tetrachlorobiphenyls	<100	<2.7	<100	<2.6
Pentachlorobiphenyls	<100	<2.7	<100	<2.6
Hexachlorobiphenyls	<200	<5.4	<200	<5.2
Heptachlorobiphenyls	<300	<8.1	<300	<7.8
Octachlorobiphenyls	<300	<8.1	<300	<7.8
Nonachlorobiphenyls	<400	<11	<400	<10
Decachlorobiphenyls	<400	<11	<400	<10
Total	---	---	1.6	0.041

TABLE 6-16. RESULTS OF THE DIOXIN AND FURAN ANALYSIS ON THE FEED MATERIAL

Compound	Detection Limit (ng/g)	Date: Time:	11/4/87 2030 (ng/g)	11/4/87 2030 DUP (ng/g)	11/5/87 2000 (ng/g)	11/6/87 1830 (ng/g)
Tetrachlorodibenzo-p-dioxin	0.068		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.28		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.50		ND	ND	ND	ND
Heptachlorodibenzo-ppdioxin	0.748		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	0.94		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.044		0.06	0.06	ND	ND
Pentachlorodibenzofurans	0.132		ND	ND	ND	ND
Hexachlorodibenzofurans	0.332		ND	ND	ND	ND
Heptachlorodibenzofurans	0.836		ND	ND	ND	ND
Octachlorodibenzofurans	2.10		ND	ND	ND	ND

Compound	Detection Limit (ng/g)	Date: Time:	11/9/87 1110 (ng/g)	11/9/87 1245 (ng/g)	11/9/87 1245 DUP (ng/g)	11/9/87 1700 (ng/g)
Tetrachlorodibenzo-p-dioxin	0.068		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.28		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.50		ND	ND	ND	ND
Heptachlorodibenzo-ppdioxin	0.748		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	0.94		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.044		ND	0.04	0.08	ND
Pentachlorodibenzofurans	0.132		ND	ND	ND	ND
Hexachlorodibenzofurans	0.332		ND	ND	ND	ND
Heptachlorodibenzofurans	0.836		ND	ND	ND	ND
Octachlorodibenzofurans	2.10		ND	ND	ND	ND

ND - not detected.

(Continued)

TABLE 6-16. (Continued)

Compound	Detection Limit (ng/g)	Date: Time:	11/10/87 1045 (ng/g)	11/10/87 1245 (ng/g)	11/10/87 1450 (ng/g)	11/10/87 1645 (ng/g)
Tetrachlorodibenzo-p-dioxin	0.068		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.28		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.50		ND	ND	ND	ND
Heptachlorodibenzo-ppdioxin	0.748		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	0.94		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.044		ND	ND	ND	ND
Pentachlorodibenzofurans	0.132		ND	ND	ND	ND
Hexachlorodibenzofurans	0.332		ND	ND	ND	ND
Heptachlorodibenzofurans	0.836		ND	ND	ND	ND
Octachlorodibenzofurans	2.10		ND	ND	ND	ND

Compound	Detection Limit (ng/g)	Date: Time:	11/11/87 1030 (ng/g)	11/11/87 1245 (ng/g)	11/11/87 1450 (ng/g)	11/11/87 1700 (ng/g)
Tetrachlorodibenzo-p-dioxin	0.068		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.28		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.50		ND	ND	ND	ND
Heptachlorodibenzo-ppdioxin	0.748		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	0.94		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.044		0.08	0.1	0.06	0.07
Pentachlorodibenzofurans	0.132		ND	ND	ND	ND
Hexachlorodibenzofurans	0.332		ND	ND	ND	ND
Heptachlorodibenzofurans	0.836		ND	ND	ND	ND
Octachlorodibenzofurans	2.10		ND	ND	ND	ND

ND - not detected.

(Continued)

TABLE 6-16. (Continued)

Compound	Detection Limit (ng/g)	Date: Time:	11/12/87 1145 (ng/g)	11/12/87 1345 (ng/g)	11/13/87 1230 (ng/g)	11/13/87 1230 DUP (ng/g)
Tetrachlorodibenzo-p-dioxin	0.068		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.28		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.50		ND	ND	ND	ND
Heptachlorodibenzo-ppdioxin	0.748		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	0.94		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.044		ND	0.05	0.05	ND
Pentachlorodibenzofurans	0.132		ND	ND	ND	ND
Hexachlorodibenzofurans	0.332		ND	ND	ND	ND
Heptachlorodibenzofurans	0.836		ND	ND	ND	ND
Octachlorodibenzofurans	2.10		ND	ND	ND	ND

ND - not detected.

TABLE 6-17. RESULTS OF THE DIOXIN AND FURAN ANALYSIS OF THE FLUE GAS+

	Primary					
Date:	11/4/87		11/5/87		11/6/87	
Time:	0938-1330		1425-1815		1035-1503	
Volume (dscm):	9.281		13.601		12.087	
Flowrate (dscmm):	1.02		1.08		1.06	
	Mass Detected (ng)	Flue Gas Conc. (ng/m^3)	Mass Detected (ng)	Flue Gas Conc. (ng/m^3)	Mass Detected (ng)	Flue Gas Conc. (ng/m^3)
2,3,7,8-TCDD	<3.0	<0.32	<3.0	<0.22	<3.0	<0.25
Total TCDD	<3.0	<0.32	<3.0	<0.22	<3.0	<0.25
Total TCDF	<2.0	<0.22	<3.0	<0.22	<2.0	<0.17
Total PCDD	<3.0	<0.32	<4.0	<0.29	<3.0	<0.25
Total PCDF	<2.0	<0.22	<3.0	<0.22	<3.0	<0.25
Total HxCDD	<4.0	<0.43	<6.0	<0.44	<5.0	<0.41
Total HxCDF	<3.0	<0.32	<4.0	<0.29	<4.0	<0.33
Total HpCDD	<5.0	<0.54	<10	<0.74	<8.0	<0.66
Total HpCDF	<3.0	<0.32	<7.0	<0.51	<5.0	<0.41
Total OCDD	<10	<1.1	<20	<1.5	<4.0	<0.33
Total OCDF	<6.0	<0.65	<20	<1.5	<18	<1.5

	Secondary					
Date:	11/4/87		11/5/87		11/6/87	
Time:	0938-1330		1400-1815		1033-1510	
Volume (dscm):	6.635		9.468		8.296	
Flowrate (dscmm):	3.29		3.31		3.64	
	Mass Detected (ng)	Flue Gas Conc. (ng/m^3)	Mass Detected (ng)	Flue Gas Conc. (ng/m^3)	Mass Detected (ng)	Flue Gas Conc. (ng/m^3)
2,3,7,8-TCDD	<6.0	<0.90	<4.0	<0.42	<3.0	<0.36
Total TCDD	<6.0	<0.90	<4.0	<0.42	<3.0	<0.36
Total TCDF	<7.0	<1.1	<7.0	<0.74	<6.0	<0.72
Total PCDD	<7.0	<1.1	<4.0	<0.42	<4.0	<0.48
Total PCDF	<6.0	<0.90	<4.0	<0.42	<3.0	<0.36
Total HxCDD	<10	<1.5	<9.0	<0.95	<6.0	<0.72
Total HxCDF	<8.0	<1.2	<7.0	<0.74	<4.0	<0.48
Total HpCDD	<20	<3.0	<20	<2.1	<10	<1.2
Total HpCDF	<10	<1.5	<20	<2.1	<6.0	<0.72
Total OCDD	<60	<9.0	<100	<11	<40	<4.8
Total OCDF	<60	<9.0	<100	<11	<20	<2.4

+ A "less than" entry defines the detection limit for that congener in that sample.

sampling system (MM5-SASS). The extracts from the filter, XAD-2 resin, resin condensate and the probe, nozzle and glassware rinses were all combined before analysis so that the PCDD/PCDF values reported represent all of the PCDD/PCDFs which passed the nozzle tip. The results are given in units of ug/m3. Each separate analysis, representing a different run, defines a column in the table. A "less than" entry defines the detection limit for that species in that sample.

Table 6-18 and 6-19 present the results of PCDD/PCDF analyses of the scrubber make-up water and scrubber liquor, respectively. Each separate analysis, representing a different day or different run, defines a column in the table. The results are reported in units of ng/L (or ppt, by weight). There was never enough of the scrubber solids to provide a sample for PCDD/PCDF analysis.

Table 6-20 presents the results of the PCDD/PCDF analyses of the furnace ash in units of ug/g (or ppm, by weight). Each separate analysis, representing a composite sample from a different day or different run, defines a column in the table.

6.5 Concentrations of the Semi-Volatile Organic Pollutants

Table 6-21 presents the results of the RCRA Appendix IX Semi-Volatile Organic Pollutants analysis (SW-846, Method 8270) of the feed material in units of µg/g (or ppm, by weight). Each separate analysis, representing a composite sample from a different day or different run, defines a column in the table.

Table 6-22 presents the results of the RCRA Appendix IX Semi-Volatile Organic Pollutants analysis of the particulate material collected by the modified EPA Method 5 source assessment sampling system (MM5-SASS). The extracts from the filter, XAD-2 resin, resin condensate and the probe, nozzle and glassware rinses were all combined before analysis so that the semi-

TABLE 6-18. RESULTS OF THE DIOXIN AND FURAN ANALYSIS ON THE SCRUBBER MAKE-UP

Compound	Detection Limit (ng/L)	Date: Time:	11/4/87 2030 (ng/L)	11/5/87 2000 (ng/L)	11/6/87 1830 (ng/L)	11/9/87 1045 (ng/L)
Tetrachlorodibenzo-p-dioxin	0.44		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	1.27		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	2.21		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	2.77		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	3.93		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.63		ND	ND	ND	ND
Pentachlorodibenzofurans	1.64		ND	ND	ND	ND
Hexachlorodibenzofurans	2.53		ND	ND	ND	ND
Heptachlorodibenzofurans	3.16		ND	ND	ND	ND
Octachlorodibenzofurans	3.95		ND	ND	ND	ND

Compound	Detection Limit (ng/L)	Date: Time:	11/10/87 1445 (ng/L)	11/11/87 1015 (ng/L)	11/12/87 1150 (ng/L)	11/13/87 1130 (ng/L)
Tetrachlorodibenzo-p-dioxin	0.44		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	1.27		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	2.21		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	2.77		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	3.93		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.63		ND	ND	ND	ND
Pentachlorodibenzofurans	1.64		ND	ND	ND	ND
Hexachlorodibenzofurans	2.53		ND	ND	ND	ND
Heptachlorodibenzofurans	3.16		ND	ND	ND	ND
Octachlorodibenzofurans	3.95		ND	ND	ND	ND

ND - not detected.

TABLE 6-19. RESULTS OF THE DIOXIN AND FURAN ANALYSIS ON THE SCRUBBER LIQUOR

Compound	Detection Limit (ng/L)	Date: Time:	11/4/87 2030 (ng/L)	11/5/87 2000 (ng/L)	11/6/87 1830 (ng/L)	11/9/87 1045 (ng/L)
Tetrachlorodibenzo-p-dioxin	0.44		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	1.27		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	2.21		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	2.27		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	3.98		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.63		ND	ND	ND	ND
Pentachlorodibenzofurans	1.64		ND	ND	ND	ND
Hexachlorodibenzofurans	2.53		ND	ND	ND	ND
Heptachlorodibenzofurans	3.16		ND	ND	ND	ND
Octachlorodibenzofurans	3.95		ND	ND	ND	ND

Compound	Detection Limit (ng/L)	Date: Time:	11/9/87 1700 (ng/L)	11/9/87 1230 (ng/L)	11/10/87 1030 (ng/L)	11/10/87 1230 (ng/L)
Tetrachlorodibenzo-p-dioxin	0.44		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	1.27		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	2.21		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	2.27		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	3.93		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.63		ND	ND	ND	ND
Pentachlorodibenzofurans	1.64		ND	ND	ND	ND
Hexachlorodibenzofurans	2.53		ND	ND	ND	ND
Heptachlorodibenzofurans	3.16		ND	ND	ND	ND
Octachlorodibenzofurans	3.95		ND	ND	ND	ND

ND - not detected.

(Continued)

TABLE 6-19. (Continued)

Compound	Detection Limit (ng/L)	Date: Time:	11/10/87 1445 (ng/L)	11/11/87 1015 (ng/L)	11/11/87 1230 (ng/L)	11/11/87 1445 (ng/L)
Tetrachlorodibenzo-p-dioxin	0.44		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	1.27		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	2.21		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	2.77		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	3.93		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.63		ND	ND	ND	ND
Pentachlorodibenzofurans	1.64		ND	ND	ND	ND
Hexachlorodibenzofurans	2.53		ND	ND	ND	ND
Heptachlorodibenzofurans	3.16		ND	ND	ND	ND
Octachlorodibenzofurans	3.95		ND	ND	ND	ND

Compound	Detection Limit (ng/L)	Date: Time:	11/11/87 1445 DUP (ng/L)	11/11/87 1630 (ng/L)	11/12/87 1145 (ng/L)	11/12/87 1145 DUP (ng/L)
Tetrachlorodibenzo-p-dioxin	0.44		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	1.27		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	2.21		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	2.77		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	3.93		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.63		ND	ND	ND	ND
Pentachlorodibenzofurans	1.64		ND	ND	ND	ND
Hexachlorodibenzofurans	2.53		ND	ND	ND	ND
Heptachlorodibenzofurans	3.16		ND	ND	ND	ND
Octachlorodibenzofurans	3.95		ND	ND	ND	ND

ND - not detected.

(Continued)

TABLE 6-19. (Continued)

Compound	Detection Limit (ng/L)	Date: Time:	11/12/87 1345 (ng/L)	11/13/87 1130 (ng/L)	11/13/87 1130 DUP (ng/L)
Tetrachlorodibenzo-p-dioxin	0.44		ND	ND	ND
Pentachlorodibenzo-p-dioxin	1.27		ND	ND	ND
Hexachlorodibenzo-p-dioxin	2.21		ND	ND	ND
Heptachlorodibenzo-p-dioxin	2.77		ND	ND	ND
Octachlorodibenzo-p-dioxin	3.93		ND	ND	ND
Tetrachlorodibenzofurans	0.63		ND	ND	ND
Pentachlorodibenzofurans	1.64		ND	ND	ND
Hexachlorodibenzofurans	2.53		ND	ND	ND
Heptachlorodibenzofurans	3.16		ND	ND	ND
Octachlorodibenzofurans	3.95		ND	ND	ND

ND - not detected.

TABLE 6-20. RESULTS OF THE DIOXIN AND FURAN ANALYSIS ON THE FURNACE ASH

Compound	Detection Limit (ng/g)	Date: Time:	11/4/87 2030 (ng/g)	11/4/87 2030 DUP (ng/g)	11/5/87 2000 (ng/g)	11/6/87 1830 (ng/g)
Tetrachlorodibenzo-p-dioxin	0.070		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.25		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.55		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	1.41		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	2.27		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.060		ND	ND	ND	ND
Pentachlorodibenzofurans	0.16		ND	ND	ND	ND
Hexachlorodibenzofurans	0.30		ND	ND	ND	ND
Heptachlorodibenzofurans	0.56		ND	ND	ND	ND
Octachlorodibenzofurans	1.05		ND	ND	ND	ND

Compound	Detection Limit (ng/g)	Date: Time:	11/9/87 1100 (ng/g)	11/9/87 1245 (ng/g)	11/9/87 1245 DUP (ng/g)	11/9/87 1700 (ng/g)
Tetrachlorodibenzo-p-dioxin	0.070		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.25		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.55		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	1.41		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	2.27		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.060		ND	ND	ND	ND
Pentachlorodibenzofurans	0.16		ND	ND	ND	ND
Hexachlorodibenzofurans	0.30		ND	ND	ND	ND
Heptachlorodibenzofurans	0.56		ND	ND	ND	ND
Octachlorodibenzofurans	1.05		ND	ND	ND	ND

ND - not detected.

(Continued)

TABLE 6-20. (Continued)

Compound	Detection Limit (ng/g)	Date: Time:	11/10/87 1045 (ng/g)	11/10/87 1245 (ng/g)	11/10/87 1450 (ng/g)	11/10/87 1645 (ng/g)
Tetrachlorodibenzo-p-dioxin	0.070		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.25		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.55		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	1.41		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	2.27		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.060		ND	ND	ND	0.07
Pentachlorodibenzofurans	0.16		ND	ND	ND	ND
Hexachlorodibenzofurans	0.30		ND	ND	ND	ND
Heptachlorodibenzofurans	0.56		ND	ND	ND	ND
Octachlorodibenzofurans	1.05		ND	ND	ND	ND

Compound	Detection Limit (ng/g)	Date: Time:	11/11/87 1030 (ng/g)	11/11/87 1245 (ng/g)	11/11/87 1450 (ng/g)	11/11/87 1700 (ng/g)
Tetrachlorodibenzo-p-dioxin	0.070		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.25		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.55		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	1.41		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	2.27		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.060		ND	ND	ND	ND
Pentachlorodibenzofurans	0.16		ND	ND	ND	ND
Hexachlorodibenzofurans	0.30		ND	ND	ND	ND
Heptachlorodibenzofurans	0.56		ND	ND	ND	ND
Octachlorodibenzofurans	1.05		ND	ND	ND	ND

ND - not detected.

(Continued)

TABLE 6-20. (Continued)

Compound	Detection Limit (ng/g)	Date: Time:	11/12/87 1145 (ng/g)	11/12/87 1345 (ng/g)	11/13/87 1230 (ng/g)	11/13/87 1230 DUP (ng/g)
Tetrachlorodibenzo-p-dioxin	0.070		ND	ND	ND	ND
Pentachlorodibenzo-p-dioxin	0.25		ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	0.55		ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	1.41		ND	ND	ND	ND
Octachlorodibenzo-p-dioxin	2.27		ND	ND	ND	ND
Tetrachlorodibenzofurans	0.060		ND	0.3	ND	ND
Pentachlorodibenzofurans	0.16		ND	ND	ND	ND
Hexachlorodibenzofurans	0.30		ND	ND	ND	ND
Heptachlorodibenzofurans	0.56		ND	ND	ND	ND
Octachlorodibenzofurans	1.05		ND	ND	ND	ND

ND - not detected.

TABLE 6-21. RESULTS OF THE SEMI-VOLATILE ORGANICS ANALYSIS ON THE FEED MATERIAL

Parameter	Detection Limit (ug/g)+	Date: Time:	11/4/87 2030 (ug/g)	11/5/87 2000 (ug/g)	11/6/87 1830 (ug/g)
Acenaphthalene	1.0		ND	ND	ND
Acenaphthene	1.0		ND	ND	ND
Acetophenone	1.0		ND	ND	ND
2-Acetylaminofluorene	25		ND	ND	ND
4-Aminobiphenyl	25		ND	ND	ND
Aniline	2.0		ND	ND	ND
Anthracene	1.0		ND	ND	ND
Aramite	NA		NA	NA	NA
Benz(a)anthracene	1.0		ND	ND	ND
Benzenethiol	NA		NA	NA	NA
Benzidine	NA		NA	NA	NA
Benzo(a)pyrene	1.0		ND	ND	ND
Benzo(b)fluoranthene	1.0		ND	ND	ND
Benzo(gh,h,i)perylene	1.0		ND	ND	ND
Benzoic acid	NA		NA	NA	NA
Benzo(k)fluoranthene	5		ND	ND	ND
p-Benzoquinone	NA		NA	NA	NA
Benzyl alcohol	2.0		ND	ND	ND
bis(2-Chloroethoxy)ethane	1.0		ND	ND	ND
bis(2-Chloroethyl)ether	1.0		ND	ND	ND
bis(2-Chloroisopropyl)ether	1.0		ND	ND	ND
bis(2-Ethylhexyl)phthalate	1.0		ND	5.5	ND
4-Bromophenyl phenyl ether	1.0		ND	ND	ND
Butyl benzyl phthalate	1.0		ND	ND	ND
2-Sec-butyl-4,6-dinitrophenol	NA		NA	NA	NA
p-Chloroaniline	20		ND	ND	ND
Chlorobenzilate	NA		NA	NA	NA
2-Chloroethyl vinyl ether	NA		NA	NA	NA
p-Chloro-m-cresol	1.0		ND	ND	ND
2-Chloronaphthalene	1.0		ND	ND	ND
2-Chlorophenol	1.0		ND	ND	ND
4-Chlorophenyl phenyl ether	1.0		ND	ND	ND
Chrysene	1.0		ND	ND	ND
o-Cresol	1.0		ND	ND	ND
p-Cresol	1.0		ND	ND	ND
Dibenzo(a,e)pyrene	NA		NA	NA	NA
Dibenzofuran	1.0		ND	ND	ND
Dibenzo(a,h)anthracene	1.0		ND	ND	ND
Dibenzo(a,h)pyrene	NA		NA	NA	NA
Dibenzo(a,i)pyrene	NA		NA	NA	NA

(Continued)

TABLE 6-21. (Continued)

Parameter	Detection Limit (ug/g)+	Date: Time:	11/4/87 2030 (ug/g)	11/5/87 2000 (ug/g)	11/6/87 1830 (ug/g)
1,2-Dichlorobenzene	1.0		ND	ND	ND
1,3-Dichlorobenzene	1.0		ND	ND	ND
1,4-Dichlorobenzene	1.0		ND	ND	ND
3,3'-Dichlorobenzidine	2.0		ND	ND	ND
2,4-Dichlorophenol	1.0		ND	ND	ND
2,6-Dichlorophenol	1.0		ND	ND	ND
Diethylphthalate	1.0		ND	ND	ND
p-Dimethylaminoazobenzene	NA		NA	NA	NA
3,3'-Dimethoxybenzidine	NA		NA	NA	NA
7,12-Dimethylbenz(a)anthracene	2.0		ND	ND	ND
3,3'-Dimethylbenzidine	5		ND	ND	ND
2,2-Dimethylphenethylamine	25		ND	ND	ND
2,4-Dimethylphenol	1.0		ND	ND	ND
Dimethylphthalate	1.0		ND	ND	ND
1,4-Dinitrobenzene	10		ND	ND	ND
4,6-Dinitro-o-cresol	5		ND	ND	ND
2,4-Dinitrophenol	5		ND	ND	ND
2,4-Dinitrotoluene	1.0		ND	ND	ND
2,6-Dinitrotoluene	1.0		ND	ND	ND
Di-n-butylphthalate	1.0		ND	ND	ND
Di-n-octylphthalate	1.0		ND	ND	ND
Di-n-propylnitrosamine	1.0		ND	ND	ND
Diphenylamine	1.0		ND	ND	ND
1,2-Diphenylhydrazine	1.0		ND	ND	ND
Fluoranthene	1.0		ND	ND	ND
Fluorene	1.0		ND	ND	ND
Hexachlorobenzene	1.0		ND	ND	ND
Hexachlorobutadiene	1.0		ND	ND	ND
Hexachlorocyclopentadiene	1.0		ND	ND	ND
Hexachloroethane	1.0		ND	ND	ND
Hexachlorophene	NA		NA	NA	NA
Hexachloropropene	1.0		ND	ND	ND
Indeno(1,2,3-c,d)pyrene	1.0		ND	ND	ND
Isophorone	1.0		ND	ND	ND
Isosafrole	10		ND	ND	ND
Methapyrilene	NA		NA	NA	NA
3-Methylcholanthrene	10		ND	ND	ND
4,4-Methylenebis(2-chloroanilne)	NA		NA	NA	NA
Methyl methanesulfonate	2.0		ND	ND	ND
2-Methylnaphthalene	1.0		ND	ND	ND
Naphthalene	1.0		ND	ND	ND

(Continued)

TABLE 6-21. (Continued)

Parameter	Detection Limit (ug/g)+	Date: Time:	11/4/87 2030 (ug/g)	11/5/87 2000 (ug/g)	11/6/87 1830 (ug/g)
1,4-Naphthoquinone	20		ND	ND	ND
1-Naphthylamine	10		ND	ND	ND
2-Naphthylamine	10		ND	ND	ND
2-Nitroaniline	5		ND	ND	ND
3-Nitroaniline	5		ND	ND	ND
4-Nitroaniline	5		ND	ND	ND
Nitrobenzene	1.0		ND	ND	ND
2-Nitrophenol	5		ND	ND	ND
4-Nitrophenol	5		ND	ND	ND
n-Nitrosodi-n-butylamine	5		ND	ND	ND
N-Nitrosodiethylamine	5		ND	ND	ND
N-Nitrosodimethylamine	5		ND	ND	ND
N-Nitrosodiphenylamine	5		ND	ND	ND
N-Nitrosomethylethylamine	5		ND	ND	ND
N-Nitrosomorpholine	5		ND	ND	ND
N-Nitrosopiperidine	5		ND	ND	ND
N-Nitrosopyrrolidene	5		ND	ND	ND
5-Nitro-o-toluidine	5		ND	ND	ND
Pentachlorobenzene	1.0		ND	ND	ND
Pentachloroethane	1.0		ND	ND	ND
Pentachloronitrobenzene	10		ND	ND	ND
Pentachlorophenol	5		ND	ND	ND
Phenacetin	10		ND	ND	ND
Phenanthrene	1.0		ND	ND	ND
Phenol	1.0		ND	ND	ND
2-Picoline	10		ND	ND	ND
Pronamide	10		ND	ND	ND
Pyrene	1.0		ND	ND	ND
Pyridine	1.0		ND	ND	ND
Resorcinol	NA		NA	NA	NA
Safrole	10		ND	ND	ND
1,2,4,5-Tetrachlorobenzene	10		ND	ND	ND
2,3,4,6-Tetrachlorophenol	10		ND	ND	ND
1,2,4-Trichlorobenzene	1.0		ND	ND	ND
2,4,5-Trichlorophenol	5		ND	ND	ND
2,4,6-Trichlorophenol	1.0		ND	ND	ND

NA - Standard was not available; the compound was searched using an NBS library of 42,000 compounds.

+ - This detection limit is a method detection limit and reflects dilutions, interferences, etc., peculiar to these samples; instrumental detection limits are lower.

TABLE 6-22. RESULTS OF THE SEMI-VOLATILE ORGANICS ANALYSIS OF THE FLUE GAS +

	Primary			
Date:	11/5/87 *		11/6/87	
Time:	1425-1815		1035-1503	
Volume (dscm):	13.601		12.087	
Flowrate (dscmm):	1.08		1.06	
Parameter	Mass Detected (ug)	Flue Gas Conc. (ug/m3)	Mass Detected (ug)	Flue Gas Conc. (ug/m3)
Acenaphthalene	< 20	< 1.5	< 10	<0.83
Acenaphthene	< 20	< 1.5	< 10	<0.83
Acetophenone	< 20	< 1.5	< 10	<0.83
2-Acetylaminofluorene	<500	< 37	<250	< 21
4-Aminobiphenyl	<500	< 37	<250	< 21
Aniline	< 40	< 2.9	< 20	< 1.7
Anthracene	< 20	< 1.5	< 10	<0.83
Aramite	NA	NA	NA	NA
Benz(a)anthracene	< 20	< 1.5	< 10	<0.83
Benzenethiol	NA	NA	NA	NA
Benzidine	<100	< 7.4	< 50	< 4.1
Benzo(a)pyrene	< 20	< 1.5	< 10	<0.83
Benzo(b)fluoranthene	< 20	< 1.5	< 10	<0.83
Benzo(gh,h,i)perylene	< 20	< 1.5	< 10	<0.83
Benzoic acid	NA	NA	30	2.5
Benzo(k)fluoranthene	< 20	< 1.5	< 10	<0.83
p-Benzoquinone	NA	NA	NA	NA
Benzyl alcohol	< 40	< 2.9	< 20	< 1.7
bis(2-Chloroethoxy)ethane	< 20	< 1.5	< 10	<0.83
bis(2-Chloroethyl)ether	< 20	< 1.5	< 10	<0.83
bis(2-Chloroisopropyl)ether	< 20	< 1.5	< 10	<0.83
bis(2-Ethylhexyl)phthalate	44	3.2	< 10	<0.83
4-Bromophenyl phenyl ether	< 20	< 1.5	< 10	<0.83
Butyl benzyl phthalate	< 20	< 1.5	< 10	<0.83
2-Sec-butyl-4,6-dinitrophenol	NA	NA	NA	NA
p-Chloroaniline	<400	< 29	<200	< 17
Chlorobenzilate	NA	NA	NA	NA
2-Chloroethyl vinyl ether	NA	NA	NA	NA
p-Chloro-m-cresol	24	1.8	< 12	<0.99
2-Chloronaphthalene	< 20	< 1.5	< 10	<0.83
2-Chlorophenol	< 20	< 1.5	< 10	<0.83
4-Chlorophenyl phenyl ether	< 20	< 1.5	< 10	<0.83
Chrysene	< 20	< 1.5	< 10	<0.83
o-Cresol	< 20	< 1.5	< 10	<0.83
p-Cresol	< 20	< 1.5	< 10	<0.83
Dibenzo(a,e)pyrene	<100	< 7.4	< 50	< 4.1
Dibenzofuran	< 20	< 1.5	< 10	<0.83
Dibenzo(a,h)anthracene	< 20	< 1.5	< 10	<0.83
Dibenzo(a,h)pyrene	< 20	< 1.5	< 10	<0.83
Dibenzo(a,i)pyrene	<100	< 7.4	< 50	< 4.1

(Continued)

TABLE 6-22. (Continued)

	Primary			
Date:	11/5/87 *		11/6/87	
Time:	1425-1815		1035-1503	
Volume (dscm):	13.601		12.087	
Flowrate (dscmm):	1.08		1.06	
Parameter	Mass Detected (ug)	Flue Gas Conc. (ug/m3)	Mass Detected (ug)	Flue Gas Conc. (ug/m3)
1,2-Dichlorobenzene	< 20	< 1.5	< 10	<0.83
1,3-Dichlorobenzene	< 20	< 1.5	< 10	<0.83
1,4-Dichlorobenzene	< 20	< 1.5	< 10	<0.83
3,3'-Dichlorobenzidine	< 40	< 2.9	< 20	< 1.7
2,4-Dichlorophenol	< 20	< 1.5	< 10	<0.83
2,6-Dichlorophenol	< 20	< 1.5	< 10	<0.83
Diethylphthalate	420	31	<210	< 17
p-Dimethylaminoazobenzene	NA	NA	<250	< 21
3,3'-Dimethoxybenzidine	NA	NA	< 50	< 4.1
7,12-Dimethylbenz(a)anthracene	< 40	< 2.9	< 20	< 1.7
3,3'-Dimethylbenzidine	<100	< 7.4	< 50	< 4.1
2,2'-Dimethylphenethylamine	<500	< 37	<250	< 21
2,4-Dimethylphenol	< 20	< 1.5	< 10	<0.83
Dimethylphthalate	< 20	< 1.5	< 10	<0.83
1,4-Dinitrobenzene	<200	< 15	<100	< 8.3
4,6-Dinitro-o-cresol	<100	< 7.4	< 50	< 4.1
2,4-Dinitrophenol	<100	< 7.4	< 50	< 4.1
2,4-Dinitrotoluene	< 20	< 1.5	< 10	<0.83
2,6-Dinitrotoluene	< 20	< 1.5	< 10	<0.83
Di-n-butylphthalate	102	7.5	14	1.2
Di-n-octylphthalate	< 20	< 1.5	< 10	<0.83
Di-n-propylnitrosamine	< 20	< 1.5	< 10	<0.83
Diphenylamine	< 20	< 1.5	< 10	<0.83
1,2-Diphenylhydrazine	<200	< 15	< 10	<0.83
Fluoranthene	< 20	< 1.5	< 10	<0.83
Fluorene	< 20	< 1.5	< 10	<0.83
Hexachlorobenzene	< 20	< 1.5	< 10	<0.83
Hexachlorobutadiene	< 20	< 1.5	< 10	<0.83
Hexachlorocyclopentadiene	< 20	< 1.5	< 10	<0.83
Hexachloroethane	< 20	< 1.5	< 10	<0.83
Hexachlorophene	NA	NA	NA	NA
Hexachloropropene	< 20	< 1.5	< 10	<0.83
Indeno(1,2,3-c,d)pyrene	< 20	< 1.5	< 10	<0.83
Isophorone	< 20	< 1.5	< 10	<0.83
Isosafrole	<200	< 15	<100	< 8.3
Methapyrilene	NA	NA	NA	NA
3-Methylcholanthrene	<200	< 15	<100	< 8.3
4,4'-Methylenebis(2-chloroaniline)	NA	NA	<200	< 17
Methyl methanesulfonate	< 40	< 2.9	< 20	< 1.7
2-Methylnaphthalene	< 20	< 1.5	< 10	<0.83
Naphthalene	< 20	< 1.5	< 10	<0.83

(Continued)

TABLE 6-22. (Continued)

	Primary			
Date:	11/5/87 *		11/6/87	
Time:	1425-1815		1035-1503	
Volume (dscm):	13.601		12.087	
Flowrate (dscmm):	1.08		1.06	
Parameter	Mass Detected (ug)	Flue Gas Conc. (ug/m3)	Mass Detected (ug)	Flue Gas Conc. (ug/m3)
1,4-Naphthoquinone	<400	< 29	<100	< 8.3
1-Naphthylamine	<200	< 15	<100	< 8.3
2-Naphthylamine	<200	< 15	<100	< 8.3
2-Nitroaniline	<100	< 7.4	< 50	< 4.1
3-Nitroaniline	<100	< 7.4	< 50	< 4.1
4-Nitroaniline	<100	< 7.4	< 50	< 4.1
Nitrobenzene	< 20	< 1.5	< 10	<0.83
2-Nitrophenol	<100	< 7.4	< 50	< 4.1
4-Nitrophenol	<100	< 7.4	< 50	< 4.1
n-Nitrosodi-n-butylamine	<100	< 7.4	< 50	< 4.1
N-Nitrosodiethylamine	<100	< 7.4	< 50	< 4.1
N-Nitrosodimethylamine	<100	< 7.4	< 50	< 4.1
N-Nitrosodiphenylamine	<100	< 7.4	< 50	< 4.1
N-Nitrosomethylethylamine	<100	< 7.4	< 50	< 4.1
N-Nitrosomorpholine	<100	< 7.4	< 50	< 4.1
N-Nitrosopiperidine	<100	< 7.4	< 50	< 4.1
N-Nitrosopyrrolidene	<100	< 7.4	< 50	< 4.1
5-Nitro-o-toluidine	<100	< 7.4	< 50	< 4.1
Pentachlorobenzene	< 20	< 1.5	< 10	<0.83
Pentachloroethane	< 20	< 1.5	< 10	<0.83
Pentachloronitrobenzene	<200	< 15	<100	< 8.3
Pentachlorophenol	<100	< 7.4	< 50	< 4.1
Phenacetin	<200	< 15	<100	< 8.3
Phenanthrene	< 20	< 1.5	< 10	<0.83
Phenol	< 20	< 1.5	< 10	<0.83
2-Picoline	<200	< 15	<100	< 8.3
Pronamide	<200	< 15	<100	< 8.3
Pyrene	< 20	< 1.5	< 10	<0.83
Pyridine	< 20	< 1.5	< 10	<0.83
Resorcinol	NA	NA	< 10	<0.83
Safrole	<200	< 15	<100	< 8.3
1,2,4,5-Tetrachlorobenzene	<200	< 15	<100	< 8.3
2,3,4,6-Tetrachlorophenol	<200	< 15	<100	< 8.3
1,2,4-Trichlorobenzene	< 20	< 1.5	< 10	<0.83
2,4,5-Trichlorophenol	<100	< 7.4	< 50	< 4.1
2,4,6-Trichlorophenol	< 20	< 1.5	< 10	<0.83

(Continued)

TABLE 6-22. (Continued)

	Secondary					
Date:	11/4/87		11/5/87 *		11/6/87	
Time:	0938-1330		1400-1815		1033-1510	
Volume (dscm):	6.635		9.468		8.296	
Flowrate (dscmm):	3.29		3.31		3.64	
Parameter	Mass Detected (ug)	Flue Gas Conc. (ug/m3)	Mass Detected (ug)	Flue Gas Conc. (ug/m3)	Mass Detected (ug)	Flue Gas Conc. (ug/m3)
Acenaphthalene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Acenaphthene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Acetophenone	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
2-Acetylaminofluorene	<500	< 75	<500	< 53	<250	< 30
4-Aminobiphenyl	<500	< 75	<500	< 53	<250	< 30
Aniline	< 40	< 6.0	< 40	< 4.2	< 20	< 2.4
Anthracene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Aramite	NA	NA	NA	NA	NA	NA
Benz(a)anthracene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Benzenethiol	NA	NA	NA	NA	NA	NA
Benzidine	NA	NA	<100	< 11	< 50	< 6.0
Benzo(a)pyrene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Benzo(b)fluoranthene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Benzo(gh,h,i)perylene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Benzoic acid	24	3.6	104	11	40	4.8
Benzo(k)fluoranthene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
p-Benzoquinone	NA	NA	NA	NA	NA	NA
Benzyl alcohol	< 40	< 6.0	< 40	< 4.2	< 20	< 2.4
bis(2-Chloroethoxy)ethane	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
bis(2-Chloroethyl)ether	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
bis(2-Chloroisopropyl)ether	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
bis(2-Ethylhexyl)phthalate	< 20	< 3.0	22	2.3	16	1.9
4-Bromophenyl phenyl ether	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Butyl benzyl phthalate	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
2-Sec-butyl-4,6-dinitrophenol	NA	NA	NA	NA	NA	NA
p-Chloroaniline	<400	< 60	<400	< 42	<200	< 24
Chlorobenzilate	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	NA	NA	NA	NA	NA	NA
p-Chloro-m-cresol	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
2-Chloronaphthalene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
2-Chlorophenol	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
4-Chlorophenyl phenyl ether	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Chrysene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
o-Cresol	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
p-Cresol	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Dibenzo(a,e)pyrene	NA	NA	<100	< 11	< 50	< 6.0
Dibenzofuran	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Dibenzo(a,h)anthracene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Dibenzo(a,h)pyrene	NA	NA	< 20	< 2.1	< 10	< 1.2
Dibenzo(a,i)pyrene	NA	NA	<100	< 11	< 50	< 6.0

(Continued)

TABLE 6-22. (Continued)

	Secondary					
Date:	11/4/87		11/5/87*		11/6/87	
Time:	0938-1330		1400-1815		1033-1510	
Volume (dscm):	6.635		9.468		8.296	
Flowrate (dscmm):	3.29		3.31		3.64	
Parameter	Mass Detected (ug)	Flue Gas Conc. (ug/m3)	Mass Detected (ug)	Flue Gas Conc. (ug/m3)	Mass Detected (ug)	Flue Gas Conc. (ug/m3)
1,2-Dichlorobenzene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
1,3-Dichlorobenzene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
1,4-Dichlorobenzene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
3,3'-Dichlorobenzidine	< 40	< 6.0	< 40	< 4.2	< 20	< 2.4
2,4-Dichlorophenol	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
2,6-Dichlorophenol	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Diethylphthalate	158	24	< 20	< 2.1	< 10	< 1.2
p-Dimethylaminoazobenzene	NA	NA	NA	NA	<250	< 30
3,3'-Dimethoxybenzidine	NA	NA	NA	NA	< 50	< 6.0
7,12-Dimethylbenz(a)anthracene	< 40	< 6.0	< 40	< 4.2	< 20	< 2.4
3,3'-Dimethylbenzidine	<100	< 15	<100	< 11	< 50	< 6.0
2,2'-Dimethylphenethylamine	<500	< 75	<500	< 53	<250	< 30
2,4-Dimethylphenol	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Dimethylphthalate	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
1,4-Dinitrobenzene	<200	< 30	<200	< 21	<100	< 12
4,6-Dinitro-o-cresol	<100	< 15	<100	< 11	< 50	< 6.0
2,4-Dinitrophenol	<100	< 15	<100	< 11	< 50	< 6.0
2,4-Dinitrotoluene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
2,6-Dinitrotoluene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Di-n-butylphthalate	139	21	40	4.2	23	2.8
Di-n-octylphthalate	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Di-n-propylnitrosamine	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Diphenylamine	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
1,2-Diphenylhydrazine	<200	< 30	<200	< 21	< 10	< 1.2
Fluoranthene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Fluorene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Hexachlorobenzene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Hexachlorobutadiene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Hexachlorocyclopentadiene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Hexachloroethane	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Hexachlorophene	NA	NA	NA	NA	NA	NA
Hexachloropropene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Indeno(1,2,3-c,d)pyrene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Isophorone	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Isosafrole	<200	< 30	<200	< 21	<100	< 12
Methapyrilene	NA	NA	NA	NA	NA	NA
3-Methylcholanthrene	<200	< 30	<200	< 21	<100	< 12
4,4'-Methylenebis(2-chloroaniline)	NA	NA	NA	NA	<200	< 24
Methyl methanesulfonate	< 40	< 6.0	< 40	< 4.2	< 20	< 2.4
2-Methylnaphthalene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Naphthalene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2

(Continued)

TABLE 6-22. (Continued)

	Secondary					
Date:	11/4/87		11/5/87*		11/6/87	
Time:	0938-1330		1400-1815		1033-1510	
Volume (dscm):	6.635		9.468		8.296	
Flowrate (dscmm):	3.29		3.31		3.64	
Parameter	Mass Detected (ug)	Flue Gas Conc. (ug/m3)	Mass Detected (ug)	Flue Gas Conc. (ug/m3)	Mass Detected (ug)	Flue Gas Conc. (ug/m3)
1,4-Naphthoquinone	<400	< 60	<400	< 42	<100	< 12
1-Naphthylamine	<200	< 30	<200	< 21	<100	< 12
2-Naphthylamine	<200	< 30	<200	< 21	<100	< 12
2-Nitroaniline	<100	< 15	<100	< 11	< 50	< 6.0
3-Nitroaniline	<100	< 15	<100	< 11	< 50	< 6.0
4-Nitroaniline	<100	< 15	<100	< 11	< 50	< 6.0
Nitrobenzene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
2-Nitrophenol	<100	< 15	<100	< 11	< 50	< 6.0
4-Nitrophenol	<100	< 15	<100	< 11	< 50	< 6.0
n-Nitrosodi-n-butylamine	<100	< 15	<100	< 11	< 50	< 6.0
N-Nitrosodiethylamine	<100	< 15	<100	< 11	< 50	< 6.0
N-Nitrosodimethylamine	<100	< 15	<100	< 11	< 50	< 6.0
N-Nitrosodiphenylamine	<100	< 15	<100	< 11	< 50	< 6.0
N-Nitrosomethylethylamine	<100	< 15	<100	< 11	< 50	< 6.0
N-Nitrosomorpholine	<100	< 15	<100	< 11	< 50	< 6.0
N-Nitrosopiperidine	<100	< 15	<100	< 11	< 50	< 6.0
N-Nitrosopyrrolidene	<100	< 15	<100	< 11	< 50	< 6.0
5-Nitro-o-toluidine	<100	< 15	<100	< 11	< 50	< 6.0
Pentachlorobenzene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Pentachloroethane	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Pentachloronitrobenzene	<200	< 30	<200	< 21	<100	< 12
Pentachlorophenol	<100	< 15	<100	< 11	< 50	< 6.0
Phenacetin	<200	< 30	<200	< 21	<100	< 12
Phenanthrene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Phenol	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
2-Picoline	<100	< 15	<200	< 21	<100	< 12
Pronamide	<200	< 30	<200	< 21	<100	< 12
Pyrene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Pyridine	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
Resorcinol	NA	NA	NA	NA	< 10	< 1.2
Safrole	<200	< 30	<200	< 21	<100	< 12
1,2,4,5-Tetrachlorobenzene	<200	< 30	<200	< 21	<100	< 12
2,3,4,6-Tetrachlorophenol	<200	< 30	<200	< 21	<100	< 12
1,2,4-Trichlorobenzene	< 20	< 3.0	< 20	< 2.1	< 10	< 1.2
2,4,5-Trichlorophenol	<100	< 15	<100	< 11	< 50	< 6.0
2,4,6-Trichlorophenol	<100	< 15	< 20	< 2.1	< 10	< 1.2

NA: not available

+ "Less than" entries represent method detection limits for the particular compound in that sample.

* Recovery of surrogates on this sample (Table 8-24) unusually low indicating that results may be inaccurate.

volatile organic pollutant values reported represent all of that compound which passed the nozzle tip. The results are given in units of ug/m3. Each separate analysis, representing a different run, defines a column in the table.

Table 6-23 and 6-24 present the results of the RCRA Appendix IX Semi-Volatile Organic Pollutants analysis of the scrubber make-up water, and scrubber liquor, respectively. Each separate analysis, representing a different day or different run, defines a column in the table. The results are reported in units of ug/L (or ppb, by weight). There was never enough of the scrubber solids to provide a sample for the semi-volatile analysis.

Table 6-25 presents the results of the RCRA Appendix IX Semi-Volatile Organic Pollutant analysis of the furnace ash in units of ug/g (or ppm, by weight). Each separate analysis, representing a composite sample from a different day or different run, defines a column in the table.

6.6 Concentrations of the Volatile Organic Pollutants

Table 6-26 presents the results of the RCRA Appendix IX Volatile Organic Pollutants analysis (SW-846, Method 8240) of the feed material in units of ug/g (or ppm, by weight). Each separate analysis, representing a composite sample from a different day or different run, defines a column in the table.

Table 6-27 presents the results of the RCRA Appendix IX Volatile Organic Pollutants analysis of the organic resin traps and associated aqueous condensate collected by the VOST sampling system. The thermal extracts from the first Tenax resin cartridge and the second Tenax/charcoal cartridge were combined on an analytical sorbent trap and later thermally desorbed into the GC/MS for analysis (SW-846, Method 5040). This was done for at least three sets of tubes from each run during the SITE testing. The results are given in units of ug/m3. In addition, the condensate, which was collected while six

TABLE 6-23. RESULTS OF THE SEMI-VOLATILE ORGANICS ANALYSIS ON THE SCRUBBER MAKE-UP

Parameter	Detection+ Limit (ug/L)	Date: Time:	11/4/87 2030 (ug/L)	11/5/87 2000 (ug/L)	11/6/87 1830 (ug/L)
Acenaphthalene	10		ND	ND	ND
Acenaphthene	10		ND	ND	ND
Acetophenone	10		ND	ND	ND
2-Acetylaminofluorene	250		ND	ND	ND
4-Aminobiphenyl	250		ND	ND	ND
Aniline	20		ND	ND	ND
Anthracene	10		ND	ND	ND
Aramite	NA		NA	NA	NA
Benz(a)anthracene	10		ND	ND	ND
Benzenethiol	NA		NA	NA	NA
Benzidine	NA		NA	NA	NA
Benzo(a)pyrene	10		ND	ND	ND
Benzo(b)fluoranthene	10		ND	ND	ND
Benzo(gh,h,i)perylene	10		ND	ND	ND
Benzoic acid	NA		NA	NA	NA
Benzo(k)fluoranthene	10		ND	ND	ND
p-Benzoquinone	NA		NA	NA	NA
Benzyl alcohol	20		ND	ND	ND
bis(2-Chloroethoxy)ethane	10		ND	ND	ND
bis(2-Chloroethyl)ether	10		ND	ND	ND
bis(2-Chloroisopropyl)ether	10		ND	ND	ND
bis(2-Ethylhexyl)phthalate	10		ND	ND	ND
4-Bromophenyl phenyl ether	10		ND	ND	ND
Butyl benzyl phthalate	10		ND	ND	ND
2-Sec-butyl-4,6-dinitrophenol	NA		NA	NA	NA
p-Chloroaniline	200		ND	ND	ND
Chlorobenzilate	NA		NA	NA	NA
2-Chloroethyl vinyl ether	NA		NA	NA	NA
p-Chloro-m-cresol	10		ND	ND	ND
2-Chloronaphthalene	10		ND	ND	ND
2-Chlorophenol	10		ND	ND	ND
4-Chlorophenyl phenyl ether	10		ND	ND	ND
Chrysene	10		ND	ND	ND
o-Cresol	10		ND	ND	ND
p-Cresol	10		ND	ND	ND
Dibenzo(a,e)pyrene	NA		NA	NA	NA
Dibenzofuran	10		ND	ND	ND
Dibenzo(a,h)anthracene	10		ND	ND	ND
Dibenzo(a,h)pyrene	NA		NA	NA	NA
Dibenzo(a,i)pyrene	NA		NA	NA	NA

(Continued)

TABLE 6-23. (Continued)

Parameter	Detection+ Limit (ug/L)	Date: Time:	11/4/87 2030 (ug/L)	11/5/87 2000 (ug/L)	11/6/87 1830 (ug/L)
1,2-Dichlorobenzene	10		ND	ND	ND
1,3-Dichlorobenzene	10		ND	ND	ND
1,4-Dichlorobenzene	10		ND	ND	ND
3,3'-Dichlorobenzidine	20		ND	ND	ND
2,4-Dichlorophenol	10		ND	ND	ND
2,6-Dichlorophenol	10		ND	ND	ND
Diethylphthalate	10		ND	ND	ND
p-Dimethylaminoazobenzene	NA		NA	NA	NA
3,3'-Dimethoxybenzidine	NA		NA	NA	NA
7,12-Dimethylbenz(a)anthracene	20		ND	ND	ND
3,3'-Dimethylbenzidine	50		ND	ND	ND
2,2-Dimethylphenethylamine	250		ND	ND	ND
2,4-Dimethylphenol	10		ND	ND	ND
Dimethylphthalate	10		ND	ND	ND
1,4-Dinitrobenzene	100		ND	ND	ND
4,6-Dinitro-o-cresol	50		ND	ND	ND
2,4-Dinitrophenol	50		ND	ND	ND
2,4-Dinitrotoluene	10		ND	ND	ND
2,6-Dinitrotoluene	10		ND	ND	ND
Di-n-butylphthalate	10		ND	ND	ND
Di-n-octylphthalate	10		ND	ND	ND
Di-n-propylnitrosamine	10		ND	ND	ND
Diphenylamine	10		ND	ND	ND
1,2-Diphenylhydrazine	100		ND	ND	ND
Fluoranthene	10		ND	ND	ND
Fluorene	10		ND	ND	ND
Hexachlorobenzene	10		ND	ND	ND
Hexachlorobutadiene	10		ND	ND	ND
Hexachlorocyclopentadiene	10		ND	ND	ND
Hexachloroethane	10		ND	ND	ND
Hexachlorophene	NA		NA	NA	NA
Hexachloropropene	10		ND	ND	ND
Indeno(1,2,3-c,d)pyrene	10		ND	ND	ND
Isophorone	10		ND	ND	ND
Isosafrole	100		ND	ND	ND
Methapyrilene	NA		NA	NA	NA
3-Methylcholanthrene	100		ND	ND	ND
4,4-Methylenebis(2-chloroanilne)	NA		NA	NA	NA
Methyl methanesulfonate	20		ND	ND	ND
2-Methylnaphthalene	10		ND	ND	ND
Naphthalene	10		ND	ND	ND

(Continued)

TABLE 6-23. (Continued)

Parameter	Detection+ Limit (ug/L)	Date: Time:	11/4/87 2030 (ug/L)	11/5/87 2000 (ug/L)	11/6/87 1830 (ug/L)
1,4-Naphthoquinone	200		ND	ND	ND
1-Naphthylamine	100		ND	ND	ND
2-Naphthylamine	100		ND	ND	ND
2-Nitroaniline	50		ND	ND	ND
3-Nitroaniline	50		ND	ND	ND
4-Nitroaniline	50		ND	ND	ND
Nitrobenzene	10		ND	ND	ND
2-Nitrophenol	50		ND	ND	ND
4-Nitrophenol	50		ND	ND	ND
n-Nitrosodi-n-butylamine	50		ND	ND	ND
N-Nitrosodiethylamine	50		ND	ND	ND
N-Nitrosodimethylamine	50		ND	ND	ND
N-Nitrosodiphenylamine	50		ND	ND	ND
N-Nitrosomethylethylamine	50		ND	ND	ND
N-Nitrosomorpholine	50		ND	ND	ND
N-Nitrosopiperidine	50		ND	ND	ND
N-Nitrosopyrrolidene	50		ND	ND	ND
5-Nitro-o-toluidine	50		ND	ND	ND
Pentachlorobenzene	10		ND	ND	ND
Pentachloroethane	10		ND	ND	ND
Pentachloronitrobenzene	100		ND	ND	ND
Pentachlorophenol	50		ND	ND	ND
Phenacetin	100		ND	ND	ND
Phenanthrene	10		ND	ND	ND
Phenol	10		ND	ND	ND
2-Picoline	50		ND	ND	ND
Pronamide	100		ND	ND	ND
Pyrene	10		ND	ND	ND
Pyridine	10		ND	ND	ND
Resorcinol	NA		NA	NA	NA
Safrole	100		ND	ND	ND
1,2,4,5-Tetrachlorobenzene	100		ND	ND	ND
2,3,4,6-Tetrachlorophenol	100		ND	ND	ND
1,2,4-Trichlorobenzene	10		ND	ND	ND
2,4,5-Trichlorophenol	50		ND	ND	ND
2,4,6-Trichlorophenol	50		ND	ND	ND

NA - Standard was not available; the compound was searched using an NBS library of 42,000 compounds.

+ - Method detection limit.

TABLE 6-24. RESULTS OF THE SEMI-VOLATILE ORGANICS ANALYSIS ON THE SCRUBBER LIQUOR

Parameter	Detection+ Limit (ug/L)	Date: Time:	11/4/87 2030 (ug/L)	11/5/87 2000 (ug/L)	11/6/87 1830 (ug/L)
Acenaphthalene	10		ND	ND	ND
Acenaphthene	10		ND	ND	ND
Acetophenone	10		ND	ND	ND
2-Acetylaminofluorene	250		ND	ND	ND
4-Aminobiphenyl	250		ND	ND	ND
Aniline	20		ND	ND	ND
Anthracene	10		ND	ND	ND
Aramite	NA		NA	NA	NA
Benz(a)anthracene	10		ND	ND	ND
Benzenethiol	NA		NA	NA	NA
Benzidine	NA		NA	NA	NA
Benzo(a)pyrene	10		ND	ND	ND
Benzo(b)fluoranthene	10		ND	ND	ND
Benzo(gh,h,i)perylene	10		ND	ND	ND
Benzoic acid	NA		NA	NA	NA
Benzo(k)fluoranthene	10		ND	ND	ND
p-Benzoquinone	NA		NA	NA	NA
Benzyl alcohol	20		ND	ND	ND
bis(2-Chloroethoxy)ethane	10		ND	ND	ND
bis(2-Chloroethyl)ether	10		ND	ND	ND
bis(2-Chloroisopropyl)ether	10		ND	ND	ND
bis(2-Ethylhexyl)phthalate	10		ND	ND	ND
4-Bromophenyl phenyl ether	10		ND	ND	ND
Butyl benzyl phthalate	10		ND	ND	ND
2-Sec-butyl-4,6-dinitrophenol	NA		NA	NA	NA
p-Chloroaniline	200		ND	ND	ND
Chlorobenzilate	NA		NA	NA	NA
2-Chloroethyl vinyl ether	NA		NA	NA	NA
p-Chloro-m-cresol	10		ND	ND	ND
2-Chloronaphthalene	10		ND	ND	ND
2-Chlorophenol	10		ND	ND	ND
4-Chlorophenyl phenyl ether	10		ND	ND	ND
Chrysene	10		ND	ND	ND
o-Cresol	10		ND	ND	ND
p-Cresol	10		ND	ND	ND
Dibenzo(a,e)pyrene	NA		NA	NA	NA
Dibenzofuran	10		ND	ND	ND
Dibenzo(a,h)anthracene	10		ND	ND	ND
Dibenzo(a,h)pyrene	NA		NA	NA	NA
Dibenzo(a,i)pyrene	NA		NA	NA	NA

(Continued)

TABLE 6-24. (Continued)

Parameter	Detection+ Limit (ug/L)	Date: Time:	11/4/87 2030 (ug/L)	11/5/87 2000 (ug/L)	11/6/87 1830 (ug/L)
1,2-Dichlorobenzene	10		ND	ND	ND
1,3-Dichlorobenzene	10		ND	ND	ND
1,4-Dichlorobenzene	10		ND	ND	ND
3,3'-Dichlorobenzidine	20		ND	ND	ND
2,4-Dichlorophenol	10		ND	ND	ND
2,6-Dichlorophenol	10		ND	ND	ND
Diethylphthalate	10		ND	ND	ND
p-Dimethylaminoazobenzene	NA		NA	NA	NA
3,3'-Dimethoxybenzidine	NA		NA	NA	NA
7,12-Dimethylbenz(a)anthracene	20		ND	ND	ND
3,3'-Dimethylbenzidine	50		ND	ND	ND
2,2-Dimethylphenethylamine	250		ND	ND	ND
2,4-Dimethylphenol	10		ND	ND	ND
Dimethylphthalate	10		ND	ND	ND
1,4-Dinitrobenzene	100		ND	ND	ND
4,6-Dinitro-o-cresol	50		ND	ND	ND
2,4-Dinitrophenol	50		ND	ND	ND
2,4-Dinitrotoluene	10		ND	ND	ND
2,6-Dinitrotoluene	10		ND	ND	ND
Di-n-butylphthalate	10		ND	ND	ND
Di-n-octylphthalate	10		ND	ND	ND
Di-n-propylnitrosamine	10		ND	ND	ND
Diphenylamine	10		ND	ND	ND
1,2-Diphenylhydrazine	100		ND	ND	ND
Fluoranthene	10		ND	ND	ND
Fluorene	10		ND	ND	ND
Hexachlorobenzene	10		ND	ND	ND
Hexachlorobutadiene	10		ND	ND	ND
Hexachlorocyclopentadiene	10		ND	ND	ND
Hexachloroethane	10		ND	ND	ND
Hexachlorophene	NA		NA	NA	NA
Hexachloropropene	10		ND	ND	ND
Indeno(1,2,3-c,d)pyrene	10		ND	ND	ND
Isophorone	10		ND	ND	ND
Isosafrole	100		ND	ND	ND
Methapyrilene	NA		NA	NA	NA
3-Methylcholanthrene	100		ND	ND	ND
4,4-Methylenebis(2-chloroanilne)	NA		NA	NA	NA
Methyl methanesulfonate	20		ND	ND	ND
2-Methylnaphthalene	10		ND	ND	ND
Naphthalene	10		ND	ND	ND

(Continued)

TABLE 6-24. (Continued)

Parameter	Detection+ Limit (ug/L)	Date: Time:	11/4/87 2030 (ug/L)	11/5/87 2000 (ug/L)	11/6/87 1830 (ug/L)
1,4-Naphthoquinone	200		ND	ND	ND
1-Naphthylamine	100		ND	ND	ND
2-Naphthylamine	100		ND	ND	ND
2-Nitroaniline	50		ND	ND	ND
3-Nitroaniline	50		ND	ND	ND
4-Nitroaniline	50		ND	ND	ND
Nitrobenzene	10		ND	ND	ND
2-Nitrophenol	50		ND	ND	ND
4-Nitrophenol	50		ND	ND	ND
n-Nitrosodi-n-butylamine	50		ND	ND	ND
N-Nitrosodiethylamine	50		ND	ND	ND
N-Nitrosodimethylamine	50		ND	ND	ND
N-Nitrosodiphenylamine	50		ND	ND	ND
N-Nitrosomethylethylamine	50		ND	ND	ND
N-Nitrosomorpholine	50		ND	ND	ND
N-Nitrosopiperidine	50		ND	ND	ND
N-Nitrosopyrrolidene	50		ND	ND	ND
5-Nitro-o-toluidine	50		ND	ND	ND
Pentachlorobenzene	10		ND	ND	ND
Pentachloroethane	10		ND	ND	ND
Pentachloronitrobenzene	100		ND	ND	ND
Pentachlorophenol	50		ND	ND	ND
Phenacetin	100		ND	ND	ND
Phenanthrene	10		ND	ND	ND
Phenol	10		ND	ND	ND
2-Picoline	50		ND	ND	ND
Pronamide	100		ND	ND	ND
Pyrene	10		ND	ND	ND
Pyridine	10		ND	ND	ND
Resorcinol	NA		NA	NA	NA
Safrole	100		ND	ND	ND
1,2,4,5-Tetrachlorobenzene	100		ND	ND	ND
2,3,4,6-Tetrachlorophenol	100		ND	ND	ND
1,2,4-Trichlorobenzene	10		ND	ND	ND
2,4,5-Trichlorophenol	50		ND	ND	ND
2,4,6-Trichlorophenol	50		ND	ND	ND

NA - Standard was not available; the compound was searched using an NBS library of 42,000 compounds.

+ - Method detection limit.

TABLE 6-25. RESULTS OF THE SEMI-VOLATILE ORGANICS ANALYSIS ON THE FURNACE ASH

Parameter	Detection+ Limit (ug/g)	Date: Time:	11/4/87 2030 (ug/g)	11/5/87 2000 (ug/g)	11/6/87 1830 (ug/g)
Acenaphthalene	1.0		ND	ND	ND
Acenaphthene	1.0		ND	ND	ND
Acetophenone	1.0		ND	ND	ND
2-Acetylaminofluorene	25		ND	ND	ND
4-Aminobiphenyl	25		ND	ND	ND
Aniline	2.0		ND	ND	ND
Anthracene	1.0		ND	ND	ND
Aramite	NA		NA	NA	NA
Benz(a)anthracene	1.0		ND	ND	ND
Benzenethiol	NA		NA	NA	NA
Benzidine	NA		NA	NA	NA
Benzo(a)pyrene	1.0		ND	ND	ND
Benzo(b)fluoranthene	1.0		ND	ND	ND
Benzo(gh,h,i)perylene	1.0		ND	ND	ND
Benzoic acid	NA		NA	NA	NA
Benzo(k)fluoranthene	1.0		ND	ND	ND
p-Benzoquinone	NA		NA	NA	NA
Benzyl alcohol	2.0		ND	ND	ND
bis(2-Chloroethoxy)ethane	1.0		ND	ND	ND
bis(2-Chloroethyl)ether	1.0		ND	ND	ND
bis(2-Chloroisopropyl)ether	1.0		ND	ND	ND
bis(2-Ethylhexyl)phthalate	1.0		ND	ND	ND
4-Bromophenyl phenyl ether	1.0		ND	ND	ND
butyl benzyl phthalate	1.0		ND	ND	ND
2-Sec-butyl-4,6-dinitrophenol	NA		NA	NA	NA
p-Chloroaniline	20		ND	ND	ND
Chlorobenzilate	NA		NA	NA	NA
2-Chloroethyl vinyl ether	NA		NA	NA	NA
p-Chloro-m-cresol	1.0		ND	ND	ND
2-Chloronaphthalene	1.0		ND	ND	ND
2-Chlorophenol	1.0		ND	ND	ND
4-Chlorophenyl phenyl ether	1.0		ND	ND	ND
Chrysene	1.0		ND	ND	ND
o-Cresol	1.0		ND	ND	ND
p-Cresol	1.0		ND	ND	ND
Dibenzo(a,e)pyrene	NA		NA	NA	NA
Dibenzofuran	1.0		ND	ND	ND
Dibenzo(a,h)anthracene	1.0		ND	ND	ND
Dibenzo(a,h)pyrene	NA		NA	NA	NA
Dibenzo(a,i)pyrene	NA		NA	NA	NA

(Continued)

TABLE 6-25. (Continued)

Parameter	Detection+ Limit (ug/g)	Date: Time:	11/4/87 2030 (ug/g)	11/5/87 2000 (ug/g)	11/6/87 1830 (ug/g)
1,2-Dichlorobenzene	1.0		ND	ND	ND
1,3-Dichlorobenzene	1.0		ND	ND	ND
1,4-Dichlorobenzene	1.0		ND	ND	ND
3,3'-Dichlorobenzidine	2.0		ND	ND	ND
2,4-Dichlorophenol	1.0		ND	ND	ND
2,6-Dichlorophenol	1.0		ND	ND	ND
Diethylphthalate	1.0		ND	ND	ND
p-Dimethylaminoazobenzene	NA		NA	NA	NA
3,3'-Dimethoxybenzidine	NA		NA	NA	NA
7,12-Dimethylbenz(a)anthracene	2.0		ND	ND	ND
3,3'-Dimethylbenzidine	5		ND	ND	ND
2,2-Dimethylphenethylamine	25		ND	ND	ND
2,4-Dimethylphenol	1.0		ND	ND	ND
Dimethylphthalate	1.0		ND	ND	ND
1,4-Dinitrobenzene	10		ND	ND	ND
4,6-Dinitro-o-cresol	5		ND	ND	ND
2,4-Dinitrophenol	5		ND	ND	ND
2,4-Dinitrotoluene	1.0		ND	ND	ND
2,6-Dinitrotoluene	1.0		ND	ND	ND
Di-n-butylphthalate	1.0		ND	ND	ND
Di-n-octylphthalate	1.0		ND	ND	ND
Di-n-propylnitrosamine	1.0		ND	ND	ND
Diphenylamine	1.0		ND	ND	ND
1,2-Diphenylhydrazine	1.0		ND	ND	ND
Fluoranthene	1.0		ND	ND	ND
Fluorene	1.0		ND	ND	ND
Hexachlorobenzene	1.0		ND	ND	ND
Hexachlorobutadiene	1.0		ND	ND	ND
Hexachlorocyclopentadiene	1.0		ND	ND	ND
Hexachloroethane	1.0		ND	ND	ND
Hexachlorophene	NA		NA	NA	NA
Hexachloropropene	1.0		ND	ND	ND
Indeno(1,2,3-c,d)pyrene	1.0		ND	ND	ND
Isophorone	1.0		ND	ND	ND
Isosafrole	10		ND	ND	ND
Methapyrilene	NA		NA	NA	NA
3-Methylcholanthrene	10		ND	ND	ND
4,4-Methylenebis(2-chloroanilne)	10		ND	ND	ND
Methyl methanesulfonate	NA		NA	NA	NA
2-Methylnaphthalene	1.0		ND	ND	ND
Naphthalene	1.0		ND	ND	ND

(Continued)

TABLE 6-25. (Continued)

Parameter	Detection+ Limit (ug/g)	Date: Time:	11/4/87 2030 (ug/g)	11/5/87 2000 (ug/g)	11/6/87 1830 (ug/g)
1,4-Naphthoquinone	10		ND	ND	ND
1-Naphthylamine	10		ND	ND	ND
2-Naphthylamine	10		ND	ND	ND
2-Nitroaniline	5		ND	ND	ND
3-Nitroaniline	5		ND	ND	ND
4-Nitroaniline	5		ND	ND	ND
Nitrobenzene	1.0		ND	ND	ND
2-Nitrophenol	5		ND	ND	ND
4-Nitrophenol	5		ND	ND	ND
n-Nitrosodi-n-butylamine	5		ND	ND	ND
N-Nitrosodiethylamine	5		ND	ND	ND
N-Nitrosodimethylamine	5		ND	ND	ND
N-Nitrosodiphenylamine	5		ND	ND	ND
N-Nitrosomethylethylamine	5		ND	ND	ND
N-Nitrosomorpholine	5		ND	ND	ND
N-Nitrosopiperidine	5		ND	ND	ND
N-Nitrosopyrrolidene	5		ND	ND	ND
5-Nitro-o-toluidine	5		ND	ND	ND
Pentachlorobenzene	1.0		ND	ND	ND
Pentachloroethane	1.0		ND	ND	ND
Pentachloronitrobenzene	10		ND	ND	ND
Pentachlorophenol	5		ND	ND	ND
Phenacetin	10		ND	ND	ND
Phenanthrene	1.0		ND	ND	ND
Phenol	1.0		ND	ND	ND
2-Picoline	10		ND	ND	ND
Pronamide	10		ND	ND	ND
Pyrene	1.0		ND	ND	ND
Pyridine	1.0		ND	ND	ND
Resorcinol	NA		NA	NA	NA
Safrole	10		ND	ND	ND
1,2,4,5-Tetrachlorobenzene	10		ND	ND	ND
2,3,4,6-Tetrachlorophenol	10		ND	ND	ND
1,2,4-Trichlorobenzene	1.0		ND	ND	ND
2,4,5-Trichlorophenol	5		ND	ND	ND
2,4,6-Trichlorophenol	1.0		ND	ND	ND

NA - Standard was not available; the compound was searched using an NBS library of 42,000 compounds.

+ - Method detection limit.

TABLE 6-26. ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN FEED MATERIAL

Date: Time:	Detection Limit	11/3/87 2030	11/4/87 2030	11/5/87 2000
Compound	(ug/g)	(ug/g)	(ug/g)	(ug/g)
Acetone	50	ND	ND	ND
Acetonitrile	50	ND	ND	ND
Acrolein	10	ND	ND	ND
Acrylonitrile	10	ND	ND	ND
Benzene	2.0	ND	ND	ND
Bromoform	2.0	ND	ND	ND
Bromomethane	2.0	ND	ND	ND
Carbon Disulfide	2.0	ND	ND	ND
Carbon Tetrachloride	2.0	ND	ND	ND
Chlorobenzene	2.0	ND	ND	ND
2-Chloro-1,3-butadiene	NA	NA	NA	NA
Chlorodibromomethane	2.0	ND	ND	ND
Chloroethane	2.0	ND	ND	ND
Chloroform	2.0	ND	ND	ND
Chloromethane	2.0	ND	ND	ND
3-Chloropropene	2.0	ND	ND	ND
cis-1,3-Dichloropropene	2.0	ND	ND	ND
1,2-Dibromo-3-chloropropane	2.0	ND	ND	ND
1,2-Dibromomethane	2.0	ND	ND	ND
Dibromomethane	2.0	ND	ND	ND
Dichlorobromomethane	2.0	ND	ND	ND
Dichlorodifluoromethane	10	ND	ND	ND
1,1-Dichloroethane	2.0	ND	ND	ND
1,2-Dichloroethane	2.0	ND	ND	ND
1,1-Dichloroethene	2.0	ND	ND	ND
1,2-Dichloropropane	2.0	ND	ND	ND
Ethyl benzene	2.0	ND	ND	ND
Ethylene oxide	NA	NA	NA	NA
Ethyl methacrylate	10	ND	ND	ND
2-Hexanone	10	ND	ND	ND
Iodomethane	10	ND	ND	ND
Methylene chloride	50	ND	ND	ND
Methyl ethyl ketone	25	32a	34b	31b
Methyl methacrylate	10	ND	ND	ND
4-Methyl-2-pentanone	10	ND	ND	ND
Propanenitrile	NA	ND	ND	ND
Styrene	2.0	ND	ND	ND
1,1,1,2-Tetrachloroethane	2.0	ND	ND	ND
1,1,2,2-Tetrachloroethane	2.0	ND	ND	ND
Tetrachloroethylene	2.0	ND	ND	ND
Toluene	2.0	ND	ND	ND
trans-1,4-Dichloro-2-butene	25	ND	ND	ND
trans-1,2-Dichloroethene	2.0	ND	ND	ND
trans-1,3-Dichloropropene	2.0	ND	ND	ND
1,1,1-Trichloroethane	2.0	ND	ND	ND
1,1,2-Trichloroethane	2.0	ND	ND	ND
Trichloroethene	2.0	17b	3.4b	ND
Trichlorofluoromethane	10	ND	ND	ND
Trichloromethanethiol	NA	NA	NA	NA
1,2,3-Trichloropropane	NA	NA	NA	NA
Vinyl acetate	25	ND	ND	ND
Vinyl chloride	2.0	ND	ND	ND
Xylenes	2.0	ND	ND	ND

ND: Not Detected
NA: Not Available due to unavailability of calibration standard
a: Solvent blank mesured 29. b: Solvent blank exceeded value in table

TABLE 6-27. CONCENTRATION OF VOLATILE ORGANIC POLLUTANTS IN THE FLUE GAS

LOCATION:	DETECTION	FIELD BLANK	PRIMARY	PRIMARY	PRIMARY
DATE:	LIMIT	11/3/87	11/3/87	11/3/87	11/3/88
TIME:	—	—	1143	1224	1303
SAMPLE:	—	AVG OF 3	PAIR 1185	PAIR 1183	PAIR 1187
	ug/m3	ug/m3	ug/m3	ug/m3	ug/m3
ACETONE	0.38	ND	ERR	27.0	ERR
ACROLEIN	0.05	ND	105.1	105.3	>649.7
ACRYLONITRILE	0.03	ND	ERR	ERR	ERR
BENZENE	0.22	0.8	>319.2A	>685.8A	>324.1A
BROMODICHLOROMETHANE	0.05	ND	ERR	ERR	ERR
BROMOFORM	0.05	ND	ERR	ERR	ERR
BROMOMETHANE	0.05	ND	19.1	7.8	13.5
2-BUTANONE	0.04	ND	3.3	ERR	20.0
CARBON DISULFIDE	0.05	ND	14.3	0.9	10.7
CARBON TETRACHLORIDE	0.14	ND	0.4	1.3	0.9
CHLOROBENZENE	0.30	ND	1.5	3.6	12.5
CHLORODIBROMOMETHANE	0.02	ND	ERR	ERR	ERR
CHLOROETHANE	0.15	ND	ERR	ERR	ERR
2-CHLOROETHYL VINYL ETHER	0.10	ND	ERR	ERR	ERR
CHLOROFORM	0.08	ND	ERR	2.5	3.4
CHLOROMETHANE	0.10	ND	>191.0	109.8	>283.1
DIBROMOMETHANE	0.05	ND	ERR	ERR	ERR
1,4-DICHLORO-2-BUTENE	0.05	ND	ERR	ERR	ERR
DICHLORODIFLUOROMETHANE	0.20	ND	ERR	ERR	ERR
1,1-DICHLOROETHANE	0.10	ND	ERR	ERR	ERR
1,2-DICHLOROETHANE	0.14	ND	ERR	ERR	ERR
1,1-DICHLOROETHENE	0.15	ND	ERR	ERR	ERR
TRANS-1,2-DICHLOROETHENE	0.02	ND	ERR	ERR	ERR
1,2-DICHLOROPROPANE	0.08	ND	ERR	ERR	ERR
CIS-1,3-DICHLOROPROPENE	0.10	ND	ERR	ERR	ERR
TRANS-1,3-DICHLOROPROPENE	0.02	ND	ERR	ERR	ERR
ETHANOL	0.60	ND	ERR	ERR	ERR
ETHYLBENZENE	0.36	ND	3.6	7.3	42.7
ETHYL METHACRYLATE	0.05	ND	ERR	ERR	ERR
2-HEXANONE	0.15	ND	ERR	ERR	ERR
IODOMETHANE	0.02	ND	39.2	23.3	29.2
METHYLENE CHLORIDE	0.14	1.1	5.1B	9.4B	6.3B
4-METHYL-2-PENTANONE	0.02	ND	ERR	ERR	ERR
STYRENE	0.60	ND	0.2	28.8	23.7
1,1,2,2-TETRACHLOROETHANE	0.02	ND	ERR	ERR	ERR
TETRACHLOROETHENE	0.21	ND	0.3	ERR	ERR
TOLUENE	0.30	ND	52.5	64.1	208.8
1,1,1-TRICHLOROETHANE	0.19	ND	0.7	ERR	ERR
1,1,2-TRICHLOROETHANE	0.01	ND	ERR	ERR	ERR
TRICHLOROETHENE	0.01	ND	ERR	ERR	ERR
TRICHLOROFLUOROMETHANE	0.01	ND	3.15	0.87	ERR
1,2,3-TRICHLOROPROPANE	0.01	ND	ERR	ERR	ERR
VINYL ACETATE	0.05	ND	ERR	ERR	ERR
VINYL CHLORIDE	0.25	ND	ERR	ERR	31.56
XYLENES	0.23	ND	0.67	2.01	27.84

ND: Value below detection limit ERR: No value available for calculation

>: Calibration range exceeded, but instrument still responding

A: Blank corrected (0.80) B: Blank Corrected (1.1)

(Continued)

TABLE 6-27. (Continued)+

LOCATION: DATE: TIME: SAMPLE:	DETECTION LIMIT — — ug/m3	FIELD BLANK 11/3-11/5 — AVG OF 3 ug/m3	SECONDARY 11/3/87 1140 PAIR 1164 ug/m3	SECONDARY 11/3/87 1345 PAIR 1163 ug/m3
ACETONE	0.38	ND	7.9	2.0
ACROLEIN	0.05	ND	23.7	ERR
ACRYLONITRILE	0.03	ND	ERR	ERR
BENZENE	0.22	0.8	8.1A	>95.3A
BROMODICHLOROMETHANE	0.05	ND	ERR	ERR
BROMOFORM	0.05	ND	ERR	ERR
BROMOMETHANE	0.05	ND	ERR	3.9
2-BUTANONE	0.04	ND	ERR	ERR
CARBON DISULFIDE	0.05	ND	4.4	ERR
CARBON TETRACHLORIDE	0.14	ND	0.2	ERR
CHLOROBENZENE	0.30	ND	ERR	7.1
CHLORODIBROMOMETHANE	0.02	ND	ERR	ERR
CHLOROETHANE	0.15	ND	ERR	ERR
2-CHLOROETHYL VINYL ETHER	0.10	ND	ERR	ERR
CHLOROFORM	0.08	ND	0.1	ERR
CHLOROMETHANE	0.10	ND	3.9	48.6
DIBROMOMETHANE	0.05	ND	ERR	ERR
1,4-DICHLORO-2-BUTENE	0.05	ND	ERR	ERR
DICHLORODIFLUOROMETHANE	0.20	ND	ERR	ERR
1,1-DICHLOROETHANE	0.10	ND	ERR	ERR
1,2-DICHLOROETHANE	0.14	ND	ERR	ERR
1,1-DICHLOROETHENE	0.15	ND	ERR	ERR
TRANS-1,2-DICHLOROETHENE	0.02	ND	ERR	ERR
1,2-DICHLOROPROPANE	0.08	ND	ERR	ERR
CIS-1,3-DICHLOROPROPENE	0.10	ND	ERR	ERR
TRANS-1,3-DICHLOROPROPENE	0.02	ND	ERR	ERR
ETHANOL	0.60	ND	ERR	ERR
ETHYLBENZENE	0.36	ND	0.7	ERR
ETHYL METHACRYLATE	0.05	ND	ERR	ERR
2-HEXANONE	0.15	ND	ERR	ERR
IODOMETHANE	0.02	ND	0.4	15.7
METHYLENE CHLORIDE	0.14	1.1	3.6B	2.4B
4-METHYL-2-PENTANONE	0.02	ND	ERR	ERR
STYRENE	0.60	ND	11.4	10.6
1,1,2,2-TETRACHLOROETHANE	0.02	ND	ERR	ERR
TETRACHLOROETHENE	0.21	ND	0.6	ERR
TOLUENE	0.30	ND	2.3	1.4
1,1,1-TRICHLOROETHANE	0.19	ND	0.4	ERR
1,1,2-TRICHLOROETHANE	0.01	ND	ERR	ERR
TRICHLOROETHENE	0.01	ND	ERR	ERR
TRICHLOROFLUOROMETHANE	0.01	ND	ERR	0.96
1,2,3-TRICHLOROPROPANE	0.01	ND	ERR	ERR
VINYL ACETATE	0.05	ND	ERR	ERR
VINYL CHLORIDE	0.25	ND	ERR	ERR
XYLENES	0.23	ND	1.38	ERR

ND: Value below detection limit   ERR: No value available for calculation

>: Calibration range exceeded, but instrument still responding

A: Blank corrected (0.80)   B: Blank Corrected (1.1)   (Continued)

+: Results of one tube lost in analyses

TABLE 6-27. (Continued)

LOCATION: DATE: TIME: SAMPLE:	DETECTION LIMIT — —	FIELD BLANK 11/3-11/5 — AVG OF 3	PRIMARY 11/4/87 1325 PAIR 1193	PRIMARY 11/4/87 1401 PAIR 1191	PRIMARY 11/4/88 1435 PAIR 1190
	ug/m3	ug/m3	ug/m3	ug/m3	ug/m3
ACETONE	0.38	ND	12.6	1.9	0.6
ACROLEIN	0.05	ND	6.5	ND	ND
ACRYLONITRILE	0.03	ND	ND	ND	ND
BENZENE	0.22	0.8	>246.0A	>90.7A	>192.8A
BROMODICHLOROMETHANE	0.05	ND	ND	ND	ND
BROMOFORM	0.05	ND	ND	ND	ND
BROMOMETHANE	0.05	ND	2.0	3.8	0.7
2-BUTANONE	0.04	ND	ND	ND	ND
CARBON DISULFIDE	0.05	ND	ND	ND	ND
CARBON TETRACHLORIDE	0.14	ND	ND	ND	ND
CHLOROBENZENE	0.30	ND	16.3	6.7	61.4
CHLORODIBROMOMETHANE	0.02	ND	ND	ND	ND
CHLOROETHANE	0.15	ND	ND	ND	ND
2-CHLOROETHYL VINYL ETHER	0.10	ND	ND	ND	ND
CHLOROFORM	0.08	ND	ND	ND	ND
CHLOROMETHANE	0.10	ND	20.5	46.2	21.7
DIBROMOMETHANE	0.05	ND	ND	ND	ND
1,4-DICHLORO-2-BUTENE	0.05	ND	ND	ND	ND
DICHLORODIFLUOROMETHANE	0.20	ND	ND	ND	ND
1,1-DICHLOROETHANE	0.10	ND	ND	ND	ND
1,2-DICHLOROETHANE	0.14	ND	ND	ND	ND
1,1-DICHLOROETHENE	0.15	ND	ND	ND	ND
TRANS-1,2-DICHLOROETHENE	0.02	ND	ND	ND	ND
1,2-DICHLOROPROPANE	0.08	ND	ND	ND	ND
CIS-1,3-DICHLOROPROPENE	0.10	ND	ND	ND	ND
TRANS-1,3-DICHLOROPROPENE	0.02	ND	ND	ND	ND
ETHANOL	0.60	ND	ND	ND	ND
ETHYLBENZENE	0.36	ND	0.9	ND	0.4
ETHYL METHACRYLATE	0.05	ND	ND	ND	ND
2-HEXANONE	0.15	ND	ND	ND	ND
IODOMETHANE	0.02	ND	12.1	14.9	7.1
METHYLENE CHLORIDE	0.14	1.1	1.8B	2.2B	1.6B
4-METHYL-2-PENTANONE	0.02	ND	ND	ND	ND
STYRENE	0.60	ND	15.8	10.1	11.3
1,1,2,2-TETRACHLOROETHANE	0.02	ND	ND	ND	ND
TETRACHLOROETHENE	0.21	ND	0.3	ND	ND
TOLUENE	0.30	ND	7.9	1.3	2.0
1,1,1-TRICHLOROETHANE	0.19	ND	ND	ND	ND
1,1,2-TRICHLOROETHANE	0.01	ND	ND	ND	ND
TRICHLOROETHENE	0.01	ND	ND	ND	ND
TRICHLOROFLUOROMETHANE	0.01	ND	2.0	0.9	0.7
1,2,3-TRICHLOROPROPANE	0.01	ND	ND	ND	ND
VINYL ACETATE	0.05	ND	ND	ND	ND
VINYL CHLORIDE	0.25	ND	ND	ND	3.6
XYLENES	0.23	ND	1.2	ND	ND

ND: Value below detection limit
>: Calibration range exceeded, but instrument still responding
A: Blank corrected (0.80) B: Blank Corrected (1.1)

(Continued)

TABLE 6-27. (Continued)

LOCATION: DATE: TIME: SAMPLE:	DETECTION LIMIT — —	FIELD BLANK 11/3-11/5 — AVG OF 3	SECONDARY 11/4/87 1335 PAIR 1141	SECONDARY 11/4/87 1410 PAIR 1138	SECONDARY 11/4/88 1440 PAIR 1137
	ug/m3	ug/m3	ug/m3	ug/m3	ug/m3
ACETONE	0.38	ND	0.5	ND	13.6
ACROLEIN	0.05	ND	ND	ND	ND
ACRYLONITRILE	0.03	ND	ND	ND	ND
BENZENE	0.22	0.8	4.1A	1.0A	0.9A
BROMODICHLOROMETHANE	0.05	ND	ND	ND	ND
BROMOFORM	0.05	ND	ND	ND	ND
BROMOMETHANE	0.05	ND	ND	ND	ND
2-BUTANONE	0.04	ND	ND	ND	ND
CARBON DISULFIDE	0.05	ND	14.9	9.4	8.5
CARBON TETRACHLORIDE	0.14	ND	ND	ND	ND
CHLOROBENZENE	0.30	ND	ND	ND	ND
CHLORODIBROMOMETHANE	0.02	ND	ND	ND	ND
CHLOROETHANE	0.15	ND	ND	ND	ND
2-CHLOROETHYL VINYL ETHER	0.10	ND	ND	ND	ND
CHLOROFORM	0.08	ND	ND	0.4	ND
CHLOROMETHANE	0.10	ND	ND	2.9	ND
DIBROMOMETHANE	0.05	ND	ND	ND	ND
1,4-DICHLORO-2-BUTENE	0.05	ND	ND	ND	ND
DICHLORODIFLUOROMETHANE	0.20	ND	ND	ND	ND
1,1-DICHLOROETHANE	0.10	ND	ND	ND	ND
1,2-DICHLOROETHANE	0.14	ND	ND	ND	ND
1,1-DICHLOROETHENE	0.15	ND	ND	ND	ND
TRANS-1,2-DICHLOROETHENE	0.02	ND	ND	ND	ND
1,2-DICHLOROPROPANE	0.08	ND	ND	ND	ND
CIS-1,3-DICHLOROPROPENE	0.10	ND	ND	ND	ND
TRANS-1,3-DICHLOROPTOPENE	0.02	ND	ND	ND	ND
ETHANOL	0.60	ND	ND	ND	ND
ETHYLBENZENE	0.36	ND	ND	0.2	ND
ETHYL METHACRYLATE	0.05	ND	ND	ND	ND
2-HEXANONE	0.15	ND	ND	ND	ND
IODOMETHANE	0.02	ND	ND	0.2	ND
METHYLENE CHLORIDE	0.14	1.1	4.4B	8.8B	13.5B
4-METHYL-2-PENTANONE	0.02	ND	ND	ND	ND
STYRENE	0.60	ND	ND	2.0	0.9
1,1,2,2-TETRACHLOROETHANE	0.02	ND	ND	ND	ND
TETRACHLOROETHENE	0.21	ND	ND	ND	ND
TOLUENE	0.30	ND	1.3	1.3	1.4
1,1,1-TRICHLOROETHANE	0.19	ND	ND	0.3	ND
1,1,2-TRICHLOROETHANE	0.01	ND	ND	ND	ND
TRICHLOROETHENE	0.01	ND	ND	ND	ND
TRICHLOROFLUOROMETHANE	0.01	ND	ND	ND	ND
1,2,3-TRICHLOROPROPANE	0.01	ND	ND	ND	ND
VINYL ACETATE	0.05	ND	ND	ND	ND
VINYL CHLORIDE	0.25	ND	ND	ND	ND
XYLENES	0.23	ND	1.38	0.4	0.4

ND: Value below detection limit
>: Calibration range exceeded, but instrument still responding
A: Blank corrected (0.80)　　B: Blank Corrected (1.1)

(Continued)

TABLE 6-27. (Continued)+

LOCATION: DATE: TIME: SAMPLE:	DETECTION LIMIT — —	FIELD BLANK 11/3-11/5 — AVG OF 3	PRIMARY 11/5/87 1545 PAIR 1134	PRIMARY 11/5/87 1617 PAIR 1129	PRIMARY 11/5/88 1647 PAIR 1136	PRIMARY 11/5/87 1718 PAIR 1135
	ug/m3	ug/m3	ug/m3	ug/m3	ug/m3	mg/m3
ACETONE	0.38	ND	6.9	4.8	ND	8.7
ACROLEIN	0.05	ND	ND	ND	ND	ND
ACRYLONITRILE	0.03	ND	ND	ND	ND	ND
BENZENE	0.22	0.8	58.3A	56.2A	18.2A	8.8A
BROMODICHLOROMETHANE	0.05	ND	ND	ND	ND	ND
BROMOFORM	0.05	ND	ND	ND	ND	ND
BROMOMETHANE	0.05	ND	ND	ND	ND	ND
2-BUTANONE	0.04	ND	ND	ND	ND	ND
CARBON DISULFIDE	0.05	ND	ND	ND	ND	ND
CARBON TETRACHLORIDE	0.14	ND	ND	ND	ND	ND
CHLOROBENZENE	0.30	ND	0.9	ND	ND	0.3
CHLORODIBROMOMETHANE	0.02	ND	ND	ND	ND	ND
CHLOROETHANE	0.15	ND	ND	ND	ND	ND
2-CHLOROETHYL VINYL ETHER	0.10	ND	ND	ND	ND	ND
CHLOROFORM	0.08	ND	ND	ND	ND	ND
CHLOROMETHANE	0.10	ND	18.2	19.0	ND	37.0
DIBROMOMETHANE	0.05	ND	ND	ND	ND	ND
1,4-DICHLORO-2-BUTENE	0.05	ND	ND	ND	ND	ND
DICHLORODIFLUOROMETHANE	0.20	ND	ND	ND	ND	ND
1,1-DICHLOROETHANE	0.10	ND	ND	ND	ND	ND
1,2-DICHLOROETHANE	0.14	ND	ND	ND	ND	ND
1,1-DICHLOROETHENE	0.15	ND	ND	ND	ND	ND
TRANS-1,2-DICHLOROETHENE	0.02	ND	ND	ND	ND	ND
1,2-DICHLOROPROPANE	0.08	ND	ND	ND	ND	ND
CIS-1,3-DICHLOROPROPENE	0.10	ND	ND	ND	ND	ND
TRANS-1,3-DICHLOROPROPENE	0.02	ND	ND	ND	ND	ND
ETHANOL	0.60	ND	ND	ND	ND	ND
ETHYLBENZENE	0.36	ND	ND	ND	ND	ND
ETHYL METHACRYLATE	0.05	ND	ND	ND	ND	ND
2-HEXANONE	0.15	ND	ND	ND	ND	ND
IODOMETHANE	0.02	ND	4.1	31.4	ND	4.8
METHYLENE CHLORIDE	0.14	1.1	1.3B	16.0B	0.3B	22.0B
4-METHYL-2-PENTANONE	0.02	ND	ND	ND	ND	ND
STYRENE	0.60	ND	0.5	19.0	ND	14.9
1,1,2,2-TETRACHLOROETHANE	0.02	ND	ND	ND	ND	ND
TETRACHLOROETHENE	0.21	ND	ND	ND	ND	ND
TOLUENE	0.30	ND	0.3	4.2	0.5	0.6
1,1,1-TRICHLOROETHANE	0.19	ND	ND	ND	0.2	ND
1,1,2-TRICHLOROETHANE	0.01	ND	ND	ND	ND	ND
TRICHLOROETHENE	0.01	ND	ND	ND	ND	ND
TRICHLOROFLUOROMETHANE	0.01	ND	ND	ND	ND	3.4
1,2,3-TRICHLOROPROPANE	0.01	ND	ND	ND	ND	ND
VINYL ACETATE	0.05	ND	ND	ND	ND	ND
VINYL CHLORIDE	0.25	ND	ND	ND	ND	ND
XYLENES	0.23	ND	ND	ND	ND	ND

ND: Value below detection limit
>: Calibration range exceeded, but instrument still responding
A: Blank corrected (0.80)   B: Blank Corrected (1.1)
+: One extra (4th) tube analyzed on this run

(Continued)

TABLE 6-27. (Continued)$^+$

LOCATION:	DETECTION	FIELD BLANK	SECONDARY	SECONDARY
DATE:	LIMIT	11/3-11/5	11/5/87	11/5/87
TIME:	—	—	1605	1640
SAMPLE:	—	AVG OF 3	PAIR 1173	PAIR 1176
	ug/m3	ug/m3	ug/m3	ug/m3
ACETONE	0.38	ND	ND	8.3
ACROLEIN	0.05	ND	ND	ND
ACRYLONITRILE	0.03	ND	ND	ND
BENZENE	0.22	0.8	0.7A	2.1A
BROMODICHLOROMETHANE	0.05	ND	ND	ND
BROMOFORM	0.05	ND	ND	ND
BROMOMETHANE	0.05	ND	ND	ND
2-BUTANONE	0.04	ND	ND	ND
CARBON DISULFIDE	0.05	ND	9.7	8.8
CARBON TETRACHLORIDE	0.14	ND	ND	ND
CHLOROBENZENE	0.30	ND	0.9	ND
CHLORODIBROMOMETHANE	0.02	ND	ND	ND
CHLOROETHANE	0.15	ND	ND	ND
2-CHLOROETHYL VINYL ETHER	0.10	ND	ND	ND
CHLOROFORM	0.08	ND	ND	ND
CHLOROMETHANE	0.10	ND	ND	2.7
DIBROMOMETHANE	0.05	ND	ND	ND
1,4-DICHLORO-2-BUTENE	0.05	ND	ND	ND
DICHLORODIFLUOROMETHANE	0.20	ND	ND	ND
1,1-DICHLOROETHANE	0.10	ND	ND	ND
1,2-DICHLOROETHANE	0.14	ND	ND	ND
1,1-DICHLOROETHENE	0.15	ND	ND	ND
TRANS-1,2-DICHLOROETHENE	0.02	ND	ND	ND
1,2-DICHLOROPROPANE	0.08	ND	ND	ND
CIS-1,3-DICHLOROPROPENE	0.10	ND	ND	ND
TRANS-1,3-DICHLOROPROPENE	0.02	ND	ND	ND
ETHANOL	0.60	ND	ND	ND
ETHYLBENZENE	0.36	ND	ND	0.4
ETHYL METHACRYLATE	0.05	ND	ND	ND
2-HEXANONE	0.15	ND	ND	ND
IODOMETHANE	0.02	ND	ND	ND
METHYLENE CHLORIDE	0.14	1.1	60.2B	7.7B
4-METHYL-2-PENTANONE	0.02	ND	ND	ND
STYRENE	0.60	ND	ND	2.8
1,1,2,2-TETRACHLOROETHANE	0.02	ND	ND	ND
TETRACHLOROETHENE	0.21	ND	ND	ND
TOLUENE	0.30	ND	0.6	0.9
1,1,1-TRICHLOROETHANE	0.19	ND	ND	ND
1,1,2-TRICHLOROETHANE	0.01	ND	ND	ND
TRICHLOROETHENE	0.01	ND	ND	ND
TRICHLOROFLUOROMETHANE	0.01	ND	ND	ND
1,2,3-TRICHLOROPROPANE	0.01	ND	ND	ND
VINYL ACETATE	0.05	ND	ND	ND
VINYL CHLORIDE	0.25	ND	ND	ND
XYLENES	0.23	ND	ND	0.5

ND: Value below detection limit

>: Calibration range exceeded, but instrument still responding

A: Blank corrected (0.80) B: Blank Corrected (1.1)

+: Results of one tube lost in analyses

sets of tubes were being used during the same test run, was liquid/liquid extracted, concentrated and analyzed by GC/MS (SW-846, Method 8240). The condensate showed only occasional and very small amounts of acetone, benzene and methylene chloride. These are probably solvent contaminants and the results of the condensate analyses will be included in the supplemental data appendix. Each separate analysis, representing a different set of tubes, defines a column in the table.

Table 6-28 and 6-29 present the results of the RCRA Appendix IX Volatile Organic Pollutants analysis of the scrubber make-up water and the scrubber liquor, respectively. Each separate analysis, representing a different day or different run, defines a column in the table. The results are reported in units of ug/L (or ppb, by weight).

Table 6-30 presents the results of the RCRA Appendix IX Volatile Organic Pollutant analysis of the furnace ash in units of ug/g (or ppm, by weight). Each separate analysis, representing a composite sample from a different day or different run, defines a column in the table.

6.7 Leachability Determinations

There were two similar but distinct aqueous leaching procedures that were used to determine what would leach out of solid materials under mildly acidic conditions. For liquid samples, the analyses were carried out directly on the acidified aqueous liquid. In the first extraction procedure (called EP Toxicity, SW-846, Method 1310), 100 g the sample was leached in 2000 ml of water at pH 5 (the leachate was maintained at pH 5 by the as-needed addition of acetic acid) and was open to the atmosphere; the extract was analyzed only for metals. In the second procedure, Toxicity Characteristic Leaching Procedure or TCLP, the extraction was carried out in an acetate buffer with zero headspace so that the volatile compounds would not be lost. The TCLP extracts were analyzed for metals, volatiles and semi-volatiles.

TABLE 6-28. ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN SCRUBBER MAKE-UP WATER

Date:	Detection Limit	11/3/87	11/4/87	11/5/87
Time:		2030	2030	2000
Compound	(ug/L)	(ug/L)	(ug/L)	(ug/L)
Acetone	50	ND	ND	ND
Acetonitrile	50	ND	ND	ND
Acrolein	10	ND	ND	ND
Acrylonitrile	10	ND	ND	ND
Benzene	2.0	2.0	3.9	13
Bromoform	2.0	ND	ND	ND
Bromomethane	2.0	ND	ND	ND
Carbon Disulfide	2.0	ND	ND	ND
Carbon Tetrachloride	2.0	ND	ND	ND
Chlorobenzene	2.0	ND	ND	ND
2-Chloro-1,3-butadiene	NA	NA	NA	NA
Chlorodibromomethane	2.0	ND	ND	ND
Chloroethane	2.0	ND	ND	ND
Chloroform	2.0	ND	ND	ND
Chloromethane	2.0	ND	ND	ND
3-Chloropropene	2.0	ND	ND	ND
cis-1,3-Dichloropropene	2.0	ND	ND	ND
1,2-Dibromo-3-chloropropane	2.0	ND	ND	ND
1,2-Dibromomethane	2.0	ND	ND	ND
Dibromomethane	2.0	ND	ND	ND
Dichlorobromomethane	2.0	ND	ND	ND
Dichlorodifluoromethane	10	ND	ND	ND
1,1-Dichloroethane	2.0	ND	ND	ND
1,2-Dichloroethane	2.0	ND	ND	ND
1,1-Dichloroethene	2.0	ND	ND	ND
1,2-Dichloropropane	2.0	ND	ND	ND
Ethyl benzene	2.0	ND	ND	ND
Ethylene oxide	NA	NA	NA	NA
Ethyl methacrylate	10	ND	ND	ND
2-Hexanone	10	ND	ND	ND
Iodomethane	10	ND	ND	ND
Methylene chloride	50	ND	ND	ND
Methyl ethyl ketone	25	ND	ND	ND
Methyl methacrylate	10	ND	ND	ND
4-Methyl-2-pentanone	10	ND	ND	ND
Propanenitrile	NA	ND	ND	ND
Styrene	2.0	ND	ND	ND
1,1,1,2-Tetrachloroethane	2.0	ND	ND	ND
1,1,2,2-Tetrachloroethane	2.0	ND	ND	ND
Tetrachloroethylene	2.0	ND	ND	ND
Toluene	2.0	2.4	ND	5.3
trans-1,4-Dichloro-2-butene	25	ND	ND	ND
trans-1,2-Dichloroethene	2.0	ND	ND	ND
trans-1,3-Dichloropropene	2.0	ND	ND	ND
1,1,1-Trichloroethane	2.0	ND	ND	ND
1,1,2-Trichloroethane	2.0	ND	ND	ND
Trichloroethene	2.0	ND	ND	7.1
Trichlorofluoromethane	10	ND	ND	ND
Trichloromethanethiol	NA	NA	NA	NA
1,2,3-Trichloropropane	NA	NA	NA	NA
Vinyl acetate	25	ND	ND	ND
Vinyl chloride	2.0	ND	ND	ND
Xylenes	2.0	ND	ND	ND

ND: Not Detected
NA: Not Available due to unavailability of calibration standard

TABLE 6-29. ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN SCRUBBER LIQUORS

Date: Time: Compound	Detection Limit (ug/L)	11/3/87 2030 (ug/L)	11/4/87 2030 (ug/L)	11/5/87 2000 (ug/L)
Acetone	50	ND	ND	ND
Acetonitrile	50	ND	ND	ND
Acrolein	10	ND	ND	ND
Acrylonitrile	10	ND	ND	ND
Benzene	2.0	ND	2.0	ND
Bromoform	2.0	ND	ND	ND
Bromomethane	2.0	ND	ND	ND
Carbon Disulfide	2.0	ND	ND	ND
Carbon Tetrachloride	2.0	ND	ND	ND
Chlorobenzene	2.0	ND	ND	ND
2-Chloro-1,3-butadiene	NA	NA	NA	NA
Chlorodibromomethane	2.0	ND	ND	ND
Chloroethane	2.0	ND	ND	ND
Chloroform	2.0	ND	ND	ND
Chloromethane	2.0	ND	ND	ND
3-Chloropropene	2.0	ND	ND	ND
cis-1,3-Dichloropropene	2.0	ND	ND	ND
1,2-Dibromo-3-chloropropane	2.0	ND	ND	ND
1,2-Dibromomethane	2.0	ND	ND	ND
Dibromomethane	2.0	ND	ND	ND
Dichlorobromomethane	2.0	ND	ND	ND
Dichlorodifluoromethane	10	ND	ND	ND
1,1-Dichloroethane	2.0	ND	ND	ND
1,2-Dichloroethane	2.0	ND	ND	ND
1,1-Dichloroethene	2.0	ND	ND	ND
1,2-Dichloropropane	2.0	ND	ND	ND
Ethyl benzene	2.0	ND	ND	ND
Ethylene oxide	NA	NA	NA	NA
Ethyl methacrylate	10	ND	ND	ND
2-Hexanone	10	ND	ND	ND
Iodomethane	10	ND	ND	ND
Methylene chloride	50	ND	ND	ND
Methyl ethyl ketone	25	ND	ND	ND
Methyl methacrylate	10	ND	ND	ND
4-Methyl-2-pentanone	10	ND	ND	ND
Propanenitrile	NA	ND	ND	ND
Styrene	2.0	ND	ND	ND
1,1,1,2-Tetrachloroethane	2.0	ND	ND	ND
1,1,2,2-Tetrachloroethane	2.0	ND	ND	ND
Tetrachloroethylene	2.0	ND	ND	ND
Toluene	2.0	11	ND	5.7
trans-1,4-Dichloro-2-butene	25	ND	ND	ND
trans-1,2-Dichloroethene	2.0	ND	ND	ND
trans-1,3-Dichloropropene	2.0	ND	ND	ND
1,1,1-Trichloroethane	2.0	ND	ND	ND
1,1,2-Trichloroethane	2.0	ND	ND	ND
Trichloroethene	2.0	ND	ND	ND
Trichlorofluoromethane	10	ND	ND	ND
Trichloromethanethiol	NA	NA	NA	NA
1,2,3-Trichloropropane	NA	NA	NA	NA
Vinyl acetate	25	ND	ND	ND
Vinyl chloride	2.0	ND	ND	ND
Xylenes	2.0	ND	ND	ND

ND: Not Detected
NA: Not Available due to unavailability of calibration standard

TABLE 6-30. ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN FURNACE ASH

Date: Time: Compound	Detection Limit (ug/g)	11/3/87 2030 (ug/g)	11/4/87 2030 (ug/g)	11/5/87 2000 (ug/g)
Acetone	50	ND	ND	ND
Acetonitrile	50	ND	ND	ND
Acrolein	10	ND	ND	ND
Acrylonitrile	10	ND	ND	ND
Benzene	2.0	ND	ND	ND
Bromoform	2.0	ND	ND	ND
Bromomethane	2.0	ND	ND	ND
Carbon Disulfide	2.0	ND	ND	ND
Carbon Tetrachloride	2.0	ND	ND	ND
Chlorobenzene	2.0	ND	ND	ND
2-Chloro-1,3-butadiene	NA	NA	NA	NA
Chlorodibromomethane	2.0	ND	ND	ND
Chloroethane	2.0	ND	ND	ND
Chloroform	2.0	ND	ND	ND
Chloromethane	2.0	ND	ND	ND
3-Chloropropene	2.0	ND	ND	ND
cis-1,3-Dichloropropene	2.0	ND	ND	ND
1,2-Dibromo-3-chloropropane	2.0	ND	ND	ND
1,2-Dibromomethane	2.0	ND	ND	ND
Dibromomethane	2.0	ND	ND	ND
Dichlorobromomethane	2.0	ND	ND	ND
Dichlorodifluoromethane	10	ND	ND	ND
1,1-Dichloroethane	2.0	ND	ND	ND
1,2-Dichloroethane	2.0	ND	ND	ND
1,1-Dichloroethene	2.0	ND	ND	ND
1,2-Dichloropropane	2.0	ND	ND	ND
Ethyl benzene	2.0	ND	ND	ND
Ethylene oxide	NA	NA	NA	NA
Ethyl methacrylate	10	ND	ND	ND
2-Hexanone	10	ND	ND	ND
Iodomethane	10	ND	ND	ND
Methylene chloride	50	ND	980	ND
Methyl ethyl ketone	25	36a	64b	40c
Methyl methacrylate	10	ND	ND	ND
4-Methyl-2-pentanone	10	ND	ND	ND
Propanenitrile	NA	ND	ND	ND
Styrene	2.0	ND	ND	ND
1,1,1,2-Tetrachloroethane	2.0	ND	ND	ND
1,1,2,2-Tetrachloroethane	2.0	ND	ND	ND
Tetrachloroethylene	2.0	ND	6.4	ND
Toluene	2.0	ND	ND	ND
trans-1,4-Dichloro-2-butene	25	ND	ND	ND
trans-1,2-Dichloroethene	2.0	ND	ND	ND
trans-1,3-Dichloropropene	2.0	ND	ND	ND
1,1,1-Trichloroethane	2.0	ND	ND	ND
1,1,2-Trichloroethane	2.0	ND	ND	ND
Trichloroethene	2.0	5c	3.9d	ND
Trichlorofluoromethane	10	ND	ND	ND
Trichloromethanethiol	NA	NA	NA	NA
1,2,3-Trichloropropane	NA	NA	NA	NA
Vinyl acetate	25	ND	ND	ND
Vinyl chloride	2.0	ND	ND	ND
Xylenes	2.0	ND	ND	ND

ND: Not Detected
NA: Not Available due to unavailability of calibration standard
a: Solvent blank measured 29 b: Solvent blank measured 54
c: Solvent blank exceeded measured value d: Solvent blank measured 3.8

Table 6-31 through 6-33 present the results of the EP Toxicity leaching of the feed material, scrubber liquors and the furnace ash. EP Toxicity procedures were also to be carried out on the scrubber solids (as called for in the QAPP), but there was not enough of the material available. The results are given in units of mg/L (or ug/g or ppm, by weight) of leachate. Each separate analysis, representing a different day or different run, defines a separate column in the table.

Table 6-34 through 6-36 present the results of the TCLP leaching of the feed material, scrubber liquors and the furnace ash. TCLP procedures were also to be carried out on the scrubber solids (as called for in the QAPP), but there was not enough of the material available. The results are given in units of mg/L (or ug/g or ppm, by weight) of leachate. Each TCCP leachate table is composed of three parts -- the metals, volatiles and the semivolatiles. Each separate analysis, representing a different day or different run, defines a separate column in the table.

6.8 Other Solids Characterizations

Certain analyses which are often used to characterize solids were subcontracted out to a laboratory (Huffman Laboratories Inc. of Golden, Colorado) which routinely does such bulk analyses. The results of those tests are reported in this section. The QAPP called for these types of analyses to be carried out on the scrubber solids, but insufficient sample was available to supply to Huffman Laboratories material for testing. Therefore, testing was confined to the feed material and the ash.

Tables 6-37 and 6-38 present the results of the ultimate analysis of the bulk solids for the feed material and furnace ash, respectively. Ultimate analysis is a routinely carried out by a procedure that is a combination of several ASTM methods; in general, the sample is heated and fully combusted and the off gases trapped and analyzed. The elements reported in this study were carbon (C), nitrogen (N), oxygen (O), sulfur (S), hydrogen (H), in addition to

TABLE 6-31. METALS ANALYSIS OF EP TOXICITY LEACHATE OF FEED MATERIAL

Date: Time: Element	Detection Limit (mg/L)	Regulatory Limit (mg/L)	11/3/87 2030 (mg/L)	11/3/87 2030 Dup (mg/L)	11/4/87 2030 (mg/L)	11/5/87 2000 (mg/L)	11/6/87 1830 (mg/L)
Arsenic	0.0020	5.0	ND	ND	ND	ND	ND
Barium	0.0090	100	0.83	0.96	0.93	0.98	0.86
Cadmium	0.0030	1.0	0.009 a	ND	0.007 a	ND	ND
Chromium	0.0090	5.0	0.049	0.063	0.058	0.047	0.026
Lead	0.0020	5.0	0.15 a	0.26	0.20 a	0.23 a	0.14
Mercury	0.0002	0.2	ND	ND	ND	ND	ND
Selenium	0.0030	1.0	ND	ND	ND	ND	ND
Silver	0.0090	5.0	0.020 a	ND	0.011 a	0.011 a	0.018

a: less than 5 times detection limit

(Continued)

TABLE 6-31. (Continued)

Date:	Detection	Regulatory	11/9/87	11/9/87	11/9/87	11/10/87	11/10/87	11/10/87	11/10/87
Time:	Limit	Level	1110	1245	1700	1045	1245	1450	1645
Element	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)
Arsenic	0.002	5.0	ND	ND	ND	ND	ND	ND	ND
Barium	0.009	100	0.94	0.77	0.80	0.68	0.70	0.89	0.85
Cadmium	0.003	1.0	0.004 a	0.007 a	0.006 a	ND	0.010 a	0.015	0.012 a
Chromium	0.009	5.0	0.05	0.031 a	0.09	0.026 a	0.039 a	0.054	0.046
Lead	0.002	5.0	0.05 a	0.20 a	0.068 a	ND	0.070 a	0.21 a	0.29
Mercury	0.0002	0.2	ND	ND	ND	ND	ND	ND	ND
Selenium	0.003	1.0	ND	ND b	ND	ND	ND	ND	ND
Silver	0.009	5.0	0.010 a	ND	ND	ND	0.014 a	0.010 a	0.015 a

Date:	Detection	Regulatory	11/11/87	11/11/87	11/11/87	11/11/87	11/11/87	11/12/87	11/12/87	11/13/87
Time:	Limit	Level	1030	1245	1245 Dup	1450	1700	1145	1345	1230
Element	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)
Arsenic	0.002	5.0	ND	ND	ND	ND	ND	ND	ND	ND
Barium	0.009	100	0.94	0.85	0.80	0.86	0.88	1.3	1.2	1.3
Cadmium	0.003	1.0	0.013 a	0.003 a	0.003 a	0.003 a	ND	ND	ND	0.004 a
Chromium	0.009	5.0	0.068	0.046	0.059	0.064	0.053	0.031 a	0.023 a	0.053
Lead	0.002	5.0	0.25 a	0.12 a	0.08 a	0.20 a	0.18 a	0.32	0.67	0.53
Mercury	0.0002	0.2	ND	ND	ND	ND	ND	ND	ND	ND
Selenium	0.003	1.0	ND	ND b	ND	ND	ND	ND	ND	ND
Silver	0.009	5.0	0.015 a	0.010 a	0.013 a	0.014 a	0.010 a	0.019 a	0.012 a	ND

a: Less than 5 times detection limit

b: Spike recovery not within acceptable limits

TABLE 6-32. METALS ANALYSIS OF EP TOXICITY LEACHATE OF SCRUBBER LIQUIDS

DATE: TIME: Element	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/3/87 2030 (mg/L)	11/4/87 2030 (mg/L)	11/5/87 2000 (mg/L)	11/6/87 1830 (mg/L)	11/9/87 1045 (mg/L)	11/9/87 1230 (mg/L)	11/9/87 1700 (mg/L)	11/10/87 1030 (mg/L)
Arsenic	0.002	5.0	0.032	0.022	0.034	0.026	---	---	0.013	0.012
Barium	0.009	100	0.69	0.24	0.69	0.14	---	---	0.25	0.28
Cadmium	0.003	1.0	0.011 a	0.062	0.049	ND	---	---	ND	0.005 a
Chromium	0.009	5.0	0.26	0.27	0.32	0.52	---	---	0.10	0.082
Lead	0.002	5.0	ND	ND	ND	ND	---	---	ND	ND
Mercury	0.0002	0.2	ND	ND	ND	ND	---	---	ND	ND
Selenium	0.003	1.0	0.012 a	0.024	0.027	0.024	---	---	ND	0.016
Silver	0.009	5.0	ND	ND	ND	ND	---	---	0.010	ND

DATE: TIME: Element	11/10/87 1230 (mg/L)	11/10/8 1445 (mg/L)	11/10/87 1630 (mg/L)	11/11/87 1015 (mg/L)	11/11/87 1230 (mg/L)	11/11/87 1445 (mg/L)	11/11/87 1630 (mg/L)	11/12/87 1145 (mg/L)	11/12/87 1345 (mg/L)	11/13/87 1130 (mg/L)
Arsenic	0.012	0.007 a	0.009 a	0.021	0.016	0.016	0.015	0.010 a	0.009 a	0.011
Barium	0.27	0.16	0.26	0.28	0.29	0.29	0.30	0.26	0.27	0.066
Cadmium	0.020	0.058	0.005 a	0.019	0.016	0.012 a	0.009 a	ND	ND	ND
Chromium	0.086	0.057	0.070	0.19	0.13	0.12	0.094	0.100	0.060	0.055
Lead	ND	ND	ND	0.10 a	ND	0.06 a	ND	ND	ND	ND
Mercury	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Selenium	0.026	0.016	0.006 a	0.007 a	0.007 a	ND	ND b	ND	ND	0.009 a
Silver	ND	ND	ND	0.010 a	ND	ND	ND	ND	ND	ND

a: Less than 5 times detection limit
b: Spike recovery not within acceptable limits
ND: Not Detected
---: Sample contaminated or destroyed

TABLE 6-33. METALS ANALYSIS OF EP TOXICITY LEACHATE ON FURNACE ASH+

Date: Time:	Detection Limit ·	Regulatory Level	11/3/87 2030	11/4/87 2030	11/5/87 2000	11/5/87 2000 Dup	11/6/87 1830
Element	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)
Arsenic	0.002	5.0	ND	ND	ND	ND	ND
Barium	0.009	100	0.54	0.68	0.97	0.85	0.95
Cadmium	0.003	1.0	0.011 a	0.006 a	0.008 a	0.009 a	0.011 a
Chromium	0.009	5.0	0.16	0.015 a	0.092	0.12	0.19
Lead	0.002	5.0	0.05 a	ND	0.13 a	0.25	0.28
Mercury	0.0002	0.2	ND	ND	ND	ND	ND
Selenium	0.003	1.0	ND b	ND	ND	ND	ND
Silver	0.009	5.0	0.020 a	ND	0.012 a	ND	0.020 a

a: Less than 5 times detection limit

b: Spike recovery not within acceptable limits

(Continued)

TABLE 6-33. (Continued)

Date: Time: Element	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/9/67 1100 (mg/L)	11/9/87 1245 (mg/L)	11/9/87 1700 (mg/L)	11/10/87 1045 (mg/L)	11/10/87 1245 (mg/L)	11/10/87 1450 (mg/L)	11/10/87 1645 (mg/L)
Arsenic	0.002	5.0	ND	0.002 a	ND	ND	0.003 a	0.004 a	0.003 a
Barium	0.009	100	0.70	0.76	0.75	1.1	2.1	0.86	0.3
Cadmium	0.003	1.0	0.018	0.017	0.009 a	0.005 a	ND	0.006 a	0.003 a
Chromium	0.009	5.0	ND	ND	0.32	0.025 a	0.059	0.022 a	0.009 a
Lead	0.002	5.0	4.1	0.38	ND	0.43	1.1	0.46	0.38
Mercury	0.0002	0.2	ND	ND	ND	ND	ND	ND	ND
Selenium	0.003	1.0	ND	ND	ND	ND	ND	ND	ND
Silver	0.009	5.0	0.017 a	ND	ND	0.018 a	0.017 a	0.012 a	0.021 a

Date: Time: Element	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/11/87 1030 (mg/L)	11/11/87 1245 (mg/L)	11/11/87 1450 (mg/L)	11/12/87 1145 (mg/L)	11/12/87 1345 (mg/L)	11/13/87 1230 (mg/L)
Arsenic	0.002	5.0	ND	ND	0.002 a	0.002 a	0.004 a	0.002 a
Barium	0.009	100	0.96	1.1	0.58	0.66	0.81	1.5
Cadmium	0.003	1.0	ND	0.010 a	0.003 a	0.037	0.023	ND
Chromium	0.009	5.0	0.031 a	0.20	0.027 a	0.022 a	0.015 a	0.041 a
Lead	0.002	5.0	ND	0.14 a	0.06 a	0.88	0.89	0.27
Mercury	0.0002	0.2	ND	ND	ND	ND	ND	ND
Selenium	0.003	1.0	ND	ND	ND	ND	ND	ND
Silver	0.009	5.0	0.010 a	0.015 a	0.010 a	0.012 a	0.009 a	0.018 a

a: Less than 5 times detection limit
b: Spike recovery not within acceptable limits
+: Sample for Run #13 (11/11/87 at 1630) lost in transit (broken vial)

TABLE 6-34. METALS, VOLATILES, AND SEMI-VOLATILES ANALYSIS OF TCLP LEACHATE OF FEED MATERIAL

Date: Time: Parameter	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/3/87 2030 (mg/L)	11/4/87 2030 (mg/L)	11/5/87 2000 (mg/L)	11/6/87 1830 (mg/L)
Arsenic	0.002	5.0	ND	ND	ND	ND
Barium	0.009	100	1.5	2.0	2.0	2.0
Cadmium	0.003	1.0	0.049	0.003 a	ND	0.005 a
Chromium	0.009	5.0	0.043 a	0.059	0.050	0.10
Lead	0.002	5.0	0.67	0.35	1.3	0.49
Mercury	0.0002	0.2	ND	ND	ND	ND
Selenium	0.003	1.0	ND	ND	ND	ND
Silver	0.009	5.0	0.013 a	0.021 a	0.011 a	0.017 a
Acrylonitrile	0.01	5.0	ND	ND	ND	ND
Benzene	0.002	0.07	ND	ND	ND	0.002
Carbon Disulfide	0.002	14.4	ND	ND	ND	ND
Carbon Tetrachloride	0.002	0.07	ND	ND	ND	ND
Chlorobenzene	0.002	1.4	ND	ND	ND	ND
Chloroform	0.002	0.07	ND	ND	ND	ND
1,2-Dichloroethane	0.002	0.40	ND	ND	ND	ND
1,1-Dichloroethene	0.002	0.1	ND	ND	ND	ND
Isobutanol	NA	36	NA	NA	NA	NA
Methylene Chloride	0.05	8.6	ND	ND	ND	ND
Methyl Ethyl Ketone	0.05	7.2	ND	ND	ND	ND
1,1,1,2-Tetrachloroethane	0.002	10.0	ND	ND	ND	ND
1,1,2,2-Tetrachloroethane	0.002	1.3	ND	ND	ND	ND
Tetrachloroethylene	0.002	0.1	ND	ND	ND	ND
Toluene	0.002	14.4	0.002	ND	ND	0.008
1,1,1-Trichloroethane	0.002	30	ND	ND	ND	ND
1,1,2-Trichloroethane	0.002	1.2	ND	ND	ND	ND
Trichloroethene	0.002	0.07	ND	ND	ND	ND
Vinyl Chloride	0.002	0.05	ND	ND	ND	ND
Bis(2-chloroethyl) ether	0.010	0.05		ND	ND	ND
m-Cresol	0.010	10.0		ND	ND	ND
o-Cresol	0.010	10.0		ND	ND	ND
p-Cresol	0.010	10.0		ND	ND	ND
1,2-Dichlorobenzene	0.010	4.3		ND	ND	ND
1,4-Dichlorobenzene	0.010	10.8		ND	ND	ND
2,4-Dinitrotoluene	0.010	0.13		ND	ND	ND
Hexachlorobenzene	0.010	0.13		ND	ND	ND
Hexachlorobutadiene	0.010	0.72		ND	ND	ND
Hexachloroethane	0.010	4.3		ND	ND	ND
Nitrobenzene	0.010	10.0		ND	ND	ND
Pentachlorophenol	0.050	3.6		ND	ND	ND
Phenol	0.010	14.4		ND	ND	ND
Pyridine	0.010	5.0		ND	ND	ND
2,3,4,6-Tetrachlorophenol	0.10	1.5		ND	ND	ND
2,4,5-Trichlorophenol	0.050	5.8		ND	ND	ND
2,4,6-Trichlorophenol	0.050	0.30		ND	ND	ND

(Continued)

TABLE 6-34. (Continued)

Date: Time:	Detection Limit	Regulatory Level	11/9/87 1110	11/9/87 1245	11/9/87 1700	11/10/87 1045	11/10/87 1245	11/10/87 1450	11/10/87 1645
Parameter	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)
Arsenic	0.002	5.0	0.014 a	0.012 a	0.014 a	ND	ND	ND	ND
Barium	0.009	100	1.8	2.0	1.8	1.8	2.2	1.5	1.5
Cadmium	0.003	1.0	0.007 a	0.009 a	0.011 a	0.012 a	0.008 a	0.014 a	0.013 a
Chromium	0.009	5.0	0.055	0.069	0.080	0.049	0.080	0.041 a	0.036 a
Lead	0.002	5.0	0.56	0.44	0.89	0.66	0.55	0.96	0.81
Mercury	0.0002	0.2	ND	ND	ND	ND	ND	ND	ND
Selenium	0.003	1.0	ND	ND	ND	ND	ND	ND	ND
Silver	0.009	5.0	0.022	0.014 a	0.018 a	0.031 a	0.018 a	0.014 a	0.026 a
Acrylonitrile	0.01	5.0	ND	ND	ND	ND	ND	ND	ND
Benzene	0.002	0.07	ND	ND	ND	ND	0.0047	0.0068	0.013
Carbon Disulfide	0.002	14.4	ND	ND	ND	ND	0.0037	0.0064	0.016
Carbon Tetrachloride	0.002	0.07	ND	ND	ND	ND	0.0044	0.0076	0.016
Chlorobenzene	0.002	1.4	ND	ND	ND	ND	0.0047	0.0067	0.012
Chloroform	0.002	0.07	ND	ND	ND	ND	0.0094	0.0068	0.013
1,2-Dichloroethane	0.002	0.40	ND	ND	ND	ND	0.0026	0.0044	0.0084
1,1-Dichloroethene	0.002	0.1	ND	ND	ND	ND	0.0039	0.0071	0.018
Isobutanol	NA	36	NA	NA	NA	NA	NA	NA	NA
Methylene Chloride	0.05	8.6	ND	ND	ND	0.090	ND	ND	ND
Methyl Ethyl Ketone	0.05	7.2	ND	ND	ND	ND	ND	ND	ND
1,1,1,2-Tetrachloroethane	0.002	10.0	ND	ND	ND	ND	0.0028	0.0045	0.0085
1,1,2,2-Tetrachloroethane	0.002	1.3	ND	ND	ND	ND	ND	0.0021	0.0032
Tetrachloroethylene	0.002	0.1	0.005	0.008	0.010	ND	0.0079	0.011	0.021
Toluene	0.002	14.4	ND	ND	ND	0.056	0.104	0.081	0.084
1,1,1-Trichloroethane	0.002	30	ND	ND	ND	ND	0.0041	0.0070	0.015
1,1,2-Trichloroethane	0.002	1.2	ND	ND	ND	ND	ND	0.0032	0.0060
Trichloroethene	0.002	0.07	ND	ND	ND	ND	0.0057	0.0076	0.016
Vinyl Chloride	0.002	0.05	ND	ND	ND	ND	0.0046	0.0088	0.028
Bis(2-chloroethyl) ether	0.010	0.05	ND	ND	ND	ND	ND	ND	ND
m-Cresol	0.010	10.0	ND	ND	ND	ND	ND	ND	ND
o-Cresol	0.010	10.0	ND	ND	ND	ND	ND	ND	ND
p-Cresol	0.010	10.0	ND	ND	ND	ND	ND	ND	ND
1,2-Dichlorobenzene	0.010	4.3	ND	ND	ND	ND	ND	ND	ND
1,4-Dichlorobenzene	0.010	10.8	ND	ND	ND	ND	ND	ND	ND
2,4-Dinitrotoluene	0.010	0.13	ND	ND	ND	ND	ND	ND	ND
Hexachlorobenzene	0.010	0.13	ND	ND	ND	ND	ND	ND	ND
Hexachlorobutadiene	0.010	0.72	ND	ND	ND	ND	ND	ND	ND
Hexachloroethane	0.010	4.3	ND	ND	ND	ND	ND	ND	ND
Nitrobenzene	0.010	10.0	ND	ND	ND	ND	ND	ND	ND
Pentachlorophenol	0.050	3.6	ND	ND	ND	ND	ND	ND	ND
Phenol	0.010	14.4	ND	ND	ND	ND	ND	ND	ND
Pyridine	0.010	5.0	ND	ND	ND	ND	ND	ND	ND
2,3,4,6-Tetrachlorophenol	0.10	1.5	ND	ND	ND	ND	ND	ND	ND
2,4,5-Trichlorophenol	0.050	5.8	ND	ND	ND	ND	ND	ND	ND
2,4,6-Trichlorophenol	0.050	0.30	ND	ND	ND	ND	ND	ND	ND

(Continued)

TABLE 6-34. (Continued)

Date: Time: Parameter	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/11/87 1030 (mg/L)	11/11/87 1245 (mg/L)	11/11/87 1450 (mg/L)	11/11/87 1700 (mg/L)	11/12/87 1145 (mg/L)	11/12/87 1345 (mg/L)	11/13/87 1230 (mg/L)
Arsenic	0.002	5.0	ND	ND	ND	ND	ND	ND	ND
Barium	0.009	100	1.5	1.7	1.8	1.3	2.0	1.8	1.9
Cadmium	0.003	1.0	0.007 a	0.009 a	0.008 a	0.012 a	0.006 a	ND	0.011 a
Chromium	0.009	5.0	0.053	0.056	0.064	0.042 a	0.034 a	0.051	0.030 a
Lead	0.002	5.0	0.73	0.53	0.71	0.53	7.0+	0.88	1.8
Mercury	0.0002	0.2	ND	ND	ND	ND	ND	ND	ND
Selenium	0.003	1.0	ND	ND b	ND	ND	ND	ND	ND
Silver	0.009	5.0	0.016 a	0.018 a	0.013 a	0.012 a	ND	0.011 a	0.010 a
Acrylonitrile	0.01	5.0	ND	ND	ND	ND	ND	ND	ND
Benzene	0.002	0.07	ND	0.0066	0.0078	ND	ND	ND	0.006
Carbon Disulfide	0.002	14.4	ND	ND	ND	ND	ND	0.006	ND
Carbon Tetrachloride	0.002	0.07	ND	ND	ND	ND	ND	ND	ND
Chlorobenzene	0.002	1.4	ND	ND	0.0022	ND	ND	ND	ND
Chloroform	0.002	0.07	ND	ND	ND	0.0053	ND	ND	ND
1,2-Dichloroethane	0.002	0.40	ND	ND	ND	ND	ND	ND	ND
1,1-Dichloroethene	0.002	0.1	ND	ND	ND	ND	ND	ND	ND
Isobutanol	NA	36	NA	NA	NA	NA	NA	NA	NA
Methylene Chloride	0.05	8.6	0.090	ND	ND	ND	ND	ND	ND
Methyl Ethyl Ketone	0.05	7.2	ND	ND	ND	ND	ND	ND	ND
1,1,1,2-Tetrachloroethane	0.002	10.0	ND	ND	ND	ND	ND	ND	ND
1,1,2,2-Tetrachloroethane	0.002	1.3	ND	ND	ND	ND	ND	ND	0.002
Tetrachloroethylene	0.002	0.1	ND	0.0070	ND	0.0046	0.005	0.005	ND
Toluene	0.002	14.4	0.056	0.018	0.0050	0.0024	0.006	0.003	0.15
1,1,1-Trichloroethane	0.002	30	ND	ND	ND	ND	ND	ND	ND
1,1,2-Trichloroethane	0.002	1.2	ND	0.0020	0.0068	ND	ND	ND	ND
Trichloroethene	0.002	0.07	ND	ND	ND	0.0054	0.007	ND	0.005
Vinyl Chloride	0.002	0.05	ND	ND	ND	ND	0.003	0.006	0.003
Bis(2-chloroethyl) ether	0.010	0.05	ND	ND	ND	ND	ND	ND	ND
m-Cresol	0.010	10.0	ND	ND	ND	ND	ND	ND	ND
o-Cresol	0.010	10.0	ND	ND	ND	ND	ND	ND	ND
p-Cresol	0.010	10.0	ND	ND	ND	ND	ND	ND	ND
1,2-Dichlorobenzene	0.010	4.3	ND	ND	ND	ND	ND	ND	ND
1,4-Dichlorobenzene	0.010	10.8	ND	ND	ND	ND	ND	ND	ND
2,4-Dinitrotoluene	0.010	0.13	ND	ND	ND	ND	ND	ND	ND
Hexachlorobenzene	0.010	0.13	ND	ND	ND	ND	ND	ND	ND
Hexachlorobutadiene	0.010	0.72	ND	ND	ND	ND	ND	ND	ND
Hexachloroethane	0.010	4.3	ND	ND	ND	ND	ND	ND	ND
Nitrobenzene	0.010	10.0	ND	ND	ND	ND	ND	ND	ND
Pentachlorophenol	0.050	3.6	ND	ND	ND	ND	ND	ND	ND
Phenol	0.010	14.4	ND	13	ND	ND	ND	ND	ND
Pyridine	0.010	5.0	ND	ND	ND	ND	ND	ND	ND
2,3,4,6-Tetrachlorophenol	0.10	1.5	ND	ND	ND	ND	ND	ND	ND
2,4,5-Trichlorophenol	0.050	5.8	ND	ND	ND	ND	ND	ND	ND
2,4,6-Trichlorophenol	0.050	0.30	ND	ND	ND	ND	ND	ND	ND

a: Less than 5 times detection limit

b: Spike recovery not within acceptable limits

ND: Not Detected

NA: Not Available due to unavailability of calibration standard

+: Verified upon reanalysis.

TABLE 6-35. METALS ANALYSIS OF THE TCLP LEACHATE OF SCRUBBER LIQUIDS

Date: Time: Element	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/3/87 2030 (mg/L)	11/4/87 2030 (mg/L)	11/5/87 2000 (mg/L)	11/6/87 1830 (mg/L)
Arsenic	0.002	5.0	0.032	0.023	0.035	0.027
Barium	0.009	100	0.72	0.23	0.70	0.14
Cadmium	0.003	1.0	0.013 a	0.060	0.048	ND
Chromium	0.009	5.0	0.29	0.27	0.31	0.51
Lead	0.002	5.0	ND	ND	ND	ND
Mercury	0.0002	0.2	ND	ND	ND	ND
Selenium	0.003	1.0	0.016	0.022	0.026	0.024
Silver	0.009	5.0	ND	ND	ND	ND

a: Less than 5 times detection limit

ND: Not Detected

(Continued)

TABLE 6-35. (Continued)

Date: Time: Element	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/9/87 1110 (mg/L)	11/9/87 1245 (mg/L)	11/9/87 1700 (mg/L)	11/10/87 1045 (mg/L)	11/10/87 1245 (mg/L)	11/10/87 1450 (mg/L)	11/10/87 1645 (mg/L)
Arsenic	0.002	5.0	---	---	0.012	0.012	0.012	0.007 a	0.010 a
Barium	0.009	100	---	---	0.26	0.28	0.27	0.16	0.25
Cadmium	0.003	1.0	---	---	0.007 a	ND	0.019	0.057	0.006 a
Chromium	0.009	5.0	---	---	0.11	0.063	0.075	0.062	0.075
Lead	0.002	5.0	---	---	ND	ND	ND	ND	ND
Mercury	0.0002	0.2	---	---	ND	ND	ND	ND	ND
Selenium	0.003	1.0	---	---	ND	0.016	0.026	0.009 a	0.005 a
Silver	0.009	5.0	---	---	ND	ND	ND	ND	ND

Date: Time: Element	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/11/87 1015 (mg/L)	11/11/87 1230 (mg/L)	11/11/87 1445 (mg/L)	11/11/87 1630 (mg/L)	11/12/87 1145 (mg/L)	11/12/87 1345 (mg/L)	11/13/87 1130 (mg/L)
Arsenic	0.002	5.0	0.022	0.017	0.016	0.015	0.010 a	0.009 a	0.011
Barium	0.009	100	0.27	0.29	0.29	0.31	0.28	0.27	0.066
Cadmium	0.003	1.0	0.019	0.023	0.018	0.015 a	ND	ND	0.008 a
Chromium	0.009	5.0	0.18	0.13	0.11	0.12	0.11	0.043 a	0.054
Lead	0.002	5.0	0.06 a	0.05 a	ND	0.07 a	ND	ND	ND
Mercury	0.0002	0.2	ND	ND	ND	ND	ND	ND	ND
Selenium	0.003	1.0	0.004 a	0.004 a	ND	ND	ND	ND	0.009 a
Silver	0.009	5.0	ND	ND	ND	ND	ND	ND	ND

a: Less than 5 times detection limit
ND: Not Detected

TABLE 6-36. METALS, VOLATILES, AND SEMI-VOLATILES OF TCLP LEACHATE OF FURNACE ASH$^+$

Date: Time: Parameter	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/3/87 2030 (mg/L)	11/4/87 2030 (mg/L)	11/5/87 2000 (mg/L)	11/6/87 1830 (mg/L)
Arsenic	0.002	5.0	ND	ND	ND	0.040
Barium	0.009	100	0.59	0.61	0.53	0.65
Cadmium	0.003	1.0	0.003 a	ND	ND	0.017
Chromium	0.009	5.0	0.15	0.012 a	0.049	0.31
Lead	0.002	5.0	ND	ND	0.05 a	1.8
Mercury	0.0002	0.2	ND	ND	ND	ND
Selenium	0.003	1.0	0.003 a	ND	ND	ND
Silver	0.009	5.0	0.018 a	0.016 a	0.019 a	0.030 a
Acrylonitrile	0.01	5.0	ND	ND	ND	ND
Benzene	0.002	0.07	ND	ND	ND	0.008
Carbon Disulfide	0.002	14.4	ND	ND	ND	ND
Carbon Tetrachloride	0.002	0.07	ND	ND	ND	ND
Chlorobenzene	0.002	1.4	ND	ND	ND	ND
Chloroform	0.002	0.07	0.0034	ND	ND	ND
1,2-Dichloroethane	0.002	0.40	ND	ND	ND	ND
1,1-Dichloroethene	0.002	0.1	ND	ND	ND	ND
Isobutanol	NA	36	NA	NA	NA	NA
Methylene Chloride	0.05	8.6	ND	ND	0.18	ND
Methyl Ethyl Ketone	0.05	7.2	ND	ND	ND	ND
1,1,1,2-Tetrachloroethane	0.002	10.0	ND	ND	ND	ND
1,1,2,2-Tetrachloroethane	0.002	1.3	ND	ND	ND	ND
Tetrachloroethylene	0.002	0.1	ND	ND	ND	ND
Toluene	0.002	14.4	ND	ND	ND	0.005
1,1,1-Trichloroethane	0.002	30	ND	ND	ND	ND
1,1,2-Trichloroethane	0.002	1.2	ND	ND	ND	ND
Trichloroethene	0.002	0.07	ND	ND	ND	ND
Vinyl Chloride	0.002	0.05	ND	ND	ND	ND
Bis(2-chloroethyl) ether	0.010	0.05		ND	ND	ND
m-Cresol	0.010	10.0		ND	ND	ND
o-Cresol	0.010	10.0		ND	ND	ND
p-Cresol	0.010	10.0		ND	ND	ND
1,2-Dichlorobenzene	0.010	4.3		ND	ND	ND
1,4-Dichlorobenzene	0.010	10.8		ND	ND	ND
2,4-Dinitrotoluene	0.010	0.13		ND	ND	ND
Hexachlorobenzene	0.010	0.13		ND	ND	ND
Hexachlorobutadiene	0.010	0.72		ND	ND	ND
Hexachloroethane	0.010	4.3		ND	ND	ND
Nitrobenzene	0.010	10.0		ND	ND	ND
Pentachlorophenol	0.050	3.6		ND	ND	ND
Phenol	0.010	14.4		ND	ND	ND
Pyridine	0.010	5.0		ND	ND	ND
2,3,4,6-Tetrachlorophenol	0.10	1.5		ND	ND	ND
2,4,5-Trichlorophenol	0.050	5.8		ND	ND	ND
2,4,6-Trichlorophenol	0.050	0.30		ND	ND	ND

(Continued)

TABLE 6-36. (Continued)

Date: Time: Parameter	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/9/87 1110 (mg/L)	11/9/87 1245 (mg/L)	11/9/87 1700 (mg/L)	11/10/87 1045 (mg/L)	11/10/87 1245 (mg/L)	11/10/87 1450 (mg/L)	11/10/87 1645 (mg/L)
Arsenic	0.002	5.0	0.02 a	0.02 a	0.02 a	0.03 a	0.03 a	0.02 a	0.03 a
Barium	0.009	100	0.78	0.99	0.98	0.72	0.83	0.64	1.5
Cadmium	0.003	1.0	0.053	0.052	0.012 a	0.009 a	0.010 a	0.028	0.026
Chromium	0.009	5.0	0.023 a	0.031 a	0.098	0.016 a	0.060	0.014 a	0.020 a
Lead	0.002	5.0	1.6	3.6	0.15 a	0.17 a	2.4	0.82	2.9
Mercury	0.0002	0.2	ND	ND	ND	ND	ND	ND	ND
Selenium	0.003	1.0	ND	ND	ND	ND	ND	ND	ND
Silver	0.009	5.0	0.011 a	0.015 a	0.024 a	0.014 a	0.012 a	0.011 a	ND
Acrylonitrile	0.01	5.0	ND	ND	ND	ND	ND	ND	ND
Benzene	0.002	0.07	0.004	0.002	0.003	ND	ND	0.005	0.002
Carbon Disulfide	0.002	14.4	ND	ND	ND	0.002	ND	ND	ND
Carbon Tetrachloride	0.002	0.07	ND	ND	ND	ND	ND	ND	ND
Chlorobenzene	0.002	1.4	ND	ND	ND	ND	ND	ND	ND
Chloroform	0.002	0.07	ND	ND	ND	ND	ND	ND	ND
1,2-Dichloroethane	0.002	0.40	ND	ND	ND	ND	ND	ND	ND
1,1-Dichloroethene	0.002	0.1	ND	ND	ND	ND	ND	ND	ND
Isobutanol	NA	36	NA	NA	NA	NA	NA	NA	NA
Methylene Chloride	0.05	8.6	ND	ND	ND	ND	ND	ND	ND
Methyl Ethyl Ketone	0.05	7.2	ND	ND	ND	ND	ND	0.055	ND
1,1,1,2-Tetrachloroethane	0.002	10.0	ND	ND	ND	ND	ND	ND	ND
1,1,2,2-Tetrachloroethane	0.002	1.3	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethylene	0.002	0.1	ND	ND	ND	ND	ND	ND	ND
Toluene	0.002	14.4	0.004	ND	ND	0.008	0.011	0.004	0.003
1,1,1-Trichloroethane	0.002	30	ND	ND	ND	ND	ND	ND	ND
1,1,2-Trichloroethane	0.002	1.2	ND	ND	ND	ND	ND	ND	ND
Trichloroethene	0.002	0.07	0.005	ND	ND	ND	ND	0.004	ND
Vinyl Chloride	0.002	0.05	ND	ND	ND	ND	ND	ND	ND
Bis(2-chloroethyl) ether	0.010	0.05		ND	ND	ND	ND	ND	ND
m-Cresol	0.010	10.0		ND	ND	ND	ND	ND	ND
o-Cresol	0.010	10.0		ND	ND	ND	ND	ND	ND
p-Cresol	0.010	10.0		ND	ND	ND	ND	ND	ND
1,2-Dichlorobenzene	0.010	4.3		ND	ND	ND	ND	ND	ND
1,4-Dichlorobenzene	0.010	10.8		ND	ND	ND	ND	ND	ND
2,4-Dinitrotoluene	0.010	0.13		ND	ND	ND	ND	ND	ND
Hexachlorobenzene	0.010	0.13		ND	ND	ND	ND	ND	ND
Hexachlorobutadiene	0.010	0.72		ND	ND	ND	ND	ND	ND
Hexachloroethane	0.010	4.3		ND	ND	ND	ND	ND	ND
Nitrobenzene	0.010	10.0		ND	ND	ND	ND	ND	ND
Pentachlorophenol	0.050	3.6		ND	ND	ND	ND	ND	ND
Phenol	0.010	14.4		ND	ND	ND	ND	ND	ND
Pyridine	0.010	5.0		ND	ND	ND	ND	ND	ND
2,3,4,6-Tetrachlorophenol	0.10	1.5		ND	ND	ND	ND	ND	ND
2,4,5-Trichlorophenol	0.050	5.8		ND	ND	ND	ND	ND	ND
2,4,6-Trichlorophenol	0.050	0.30		ND	ND	ND	ND	ND	ND

(Continued)

TABLE 6-36. (Continued)

Date: Time: Parameter	Detection Limit (mg/L)	Regulatory Level (mg/L)	11/11/87 1030 (mg/L)	11/11/87 1245 (mg/L)	11/11/87 1450 (mg/L)	11/12/87 1145 (mg/L)	11/12/87 1345 (mg/L)	11/13/87 1230 (mg/L)
Arsenic	0.002	5.0	0.02 a	ND	ND	ND	ND	ND
Barium	0.009	100	0.95	0.70	0.68	0.88	1.5	0.85
Cadmium	0.003	1.0	0.010 a	0.004 a	0.033	0.073	0.045	0.005 a
Chromium	0.009	5.0	ND	0.14	0.026 a	0.038 a	0.035 a	0.025 a
Lead	0.002	5.0	1.00	0.05 a	4.1	3.8	6.2	0.23 a
Mercury	0.0002	0.2	ND	ND	ND	0.0006a	ND	ND
Selenium	0.003	1.0	ND	ND	ND	ND	ND	ND
Silver	0.009	5.0	0.022 a	0.010 a	ND	0.010 a	0.014 a	0.009 a
Acrylonitrile	0.01	5.0	ND	ND	ND	ND	ND	ND
Benzene	0.002	0.07	0.003	ND	ND	0.003	0.004	ND
Carbon Disulfide	0.002	14.4	ND	ND	ND	ND	ND	ND
Carbon Tetrachloride	0.002	0.07	ND	ND	ND	ND	ND	ND
Chlorobenzene	0.002	1.4	ND	ND	ND	ND	ND	ND
Chloroform	0.002	0.07	ND	ND	ND	ND	ND	ND
1,2-Dichloroethane	0.002	0.40	ND	ND	ND	ND	ND	ND
1,1-Dichloroethene	0.002	0.1	ND	ND	ND	ND	ND	ND
Isobutanol	NA	36	NA	NA	NA	NA	NA	NA
Methylene Chloride	0.05	8.6	ND	ND	ND	ND	ND	ND
Methyl Ethyl Ketone	0.05	7.2	ND	ND	ND	ND	ND	ND
1,1,1,2-Tetrachloroethane	0.002	10.0	ND	ND	ND	ND	ND	ND
1,1,2,2-Tetrachloroethane	0.002	1.3	ND	ND	ND	0.002	ND	ND
Tetrachloroethylene	0.002	0.1	ND	ND	ND	ND	ND	ND
Toluene	0.002	14.4	0.013	ND	ND	0.013	0.009	0.022
1,1,1-Trichloroethane	0.002	30	ND	ND	ND	ND	ND	ND
1,1,2-Trichloroethane	0.002	1.2	ND	ND	ND	ND	ND	ND
Trichloroethene	0.002	0.07	ND	ND	ND	0.004	0.004	0.009
Vinyl Chloride	0.002	0.05	ND	ND	ND	0.004	0.003	0.003
Bis(2-chloroethyl) ether	0.010	0.05	ND	ND	ND	ND	ND	ND
m-Cresol	0.010	10.0	ND	ND	ND	ND	ND	ND
o-Cresol	0.010	10.0	ND	ND	ND	ND	ND	ND
p-Cresol	0.010	10.0	ND	ND	ND	ND	ND	ND
1,2-Dichlorobenzene	0.010	4.3	ND	ND	ND	ND	ND	ND
1,4-Dichlorobenzene	0.010	10.8	ND	ND	ND	ND	ND	ND
2,4-Dinitrotoluene	0.010	0.13	ND	ND	ND	ND	ND	ND
Hexachlorobenzene	0.010	0.13	ND	ND	ND	ND	ND	ND
Hexachlorobutadiene	0.010	0.72	ND	ND	ND	ND	ND	ND
Hexachloroethane	0.010	4.3	ND	ND	ND	ND	ND	ND
Nitrobenzene	0.010	10.0	ND	ND	ND	ND	ND	ND
Pentachlorophenol	0.050	3.6	ND	ND	ND	ND	ND	ND
Phenol	0.010	14.4	ND	ND	ND	ND	ND	ND
Pyridine	0.010	5.0	ND	ND	ND	ND	ND	ND
2,3,4,6-Tetrachlorophenol	0.10	1.5	ND	ND	ND	ND	ND	ND
2,4,5-Trichlorophenol	0.050	5.8	ND	ND	ND	ND	ND	ND
2,4,6-Trichlorophenol	0.050	0.30	ND	ND	ND	ND	ND	ND

a: Less than 5 times detection limit
ND: Not Detected
NA: Not Available due to unavailability of calibration standard
+: Sample for Run #13 (11/11/87 at 1630) lost in transit (broken vial)

TABLE 6-37. CHARACTERIZATION OF FEED MATERIAL (SOIL)

Run # Date	1 11/3/87	2 11/4/87	3 11/5/87	3 (DUP) 11/5/87	3 11/6/87	10 11/9/87	14 11/9/87	5 11/9/87	6 11/10/87	8 11/10/87
Moisture (%)	8.40	9.13	11.27	11.41	10.62	10.49	10.82	11.51	12.22	11.72
Carbon (%)	3.09	3.19	2.45	2.36	2.19	2.47	2.40	2.33	3.02	3.36
Hydrogen (%)	1.31	1.36	1.71	1.67	1.46	1.41	1.43	1.47	1.60	1.60
Oxygen (%)	8.94	11.82	12.39	12.94	12.27	10.95	11.59	13.47	12.81	12.76
Nitrogen (%)	0.10	0.11	0.09	0.11	0.11	0.11	0.11	0.11	0.14	0.11
Sulfur (%)	<0.01	<0.01	<0.01	<0.01	0.04	<0.01	<0.01	0.01	0.06	0.05
Chlorine (%)	0.10	0.10	0.10	0.10	0.12	0.09	0.10	0.23	0.08	0.09
Ash (%)	86.46	83.42	83.26	82.82	83.81	84.97	84.37	82.38	82.29	82.03
Density (g/cc)	2.37	2.35	2.33	2.30	2.34	2.32	2.32	2.29	2.23	2.22
HHV (BTU/Lb)	230.	183.	208.	246.	200.	229.	207.	205.	509.	548.

Run # Date	15 11/10/87	15 (DUP) 11/10/87	18 11/10/87	7 11/11/87	9 11/11/87	11 11/11/87	13 11/11/87	16 11/12/87	17 11/12/87	19 11/13/87
Moisture (%)	13.32	13.86	12.63	11.79	9.96	12.49	11.12	10.99	11.33	12.32
Carbon (%)	4.23	4.11	3.22	3.11	1.81	1.91	2.37	2.54	2.05	3.30
Hydrogen (%)	1.51	1.62	1.73	1.61	1.34	1.58	1.45	1.49	1.47	1.75
Oxygen (%)	12.36	13.11	12.82	11.58	10.64	13.15	12.03	12.10	12.34	13.20
Nitrogen (%)	0.09	0.08	0.11	0.12	0.07	0.09	0.11	0.08	0.08	0.10
Sulfur (%)	0.02	0.02	0.01	<0.01	<0.01	0.01	0.01	0.02	0.01	0.02
Chlorine (%)	0.10	0.10	0.10	0.18	0.08	0.09	0.07	0.12	0.09	0.09
Ash (%)	81.69	80.96	82.01	83.40	86.06	83.17	83.96	83.65	83.96	81.54
Density (g/cc)	2.25	2.24	2.30	2.29	2.23	2.29	2.28	2.32	2.28	2.16
HHV (BTU/Lb)	634.	609.	558.	319.	180.	194.	147.	201.	192.	667.

TABLE 6-38. CHARACTERIZATION OF FURNACE ASH

Run # Date	1 11/3/87	2 11/4/87	3 11/5/87	3 (DUP) 11/5/87	3 11/6/87	10 11/9/87	14 11/9/87	5 11/9/87	6 11/10/87	8 11/10/87
Moisture (%)	0.48	0.57	1.54	0.95	0.61	0.83	0.58	0.67	0.95	0.71
Carbon (%)	0.36	0.50	0.50	0.29	0.49	1.17	1.21	0.57	0.57	0.45
Hydrogen (%)	0.07	0.13	0.17	0.08	0.12	0.09	0.09	0.07	0.10	0.10
Oxygen (%)	<0.5	<0.5	<0.5	<0.5	<0.5	1.26	3.48	0.64	1.06	0.66
Nitrogen (%)	<0.02	<0.02	<0.02	<0.02	0.04	0.04	<0.02	<0.02	<0.02	<0.02
Sulfur (%)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.05
Chlorine (%)	0.05	0.05	0.05	0.05	0.04	0.04	0.06	0.05	0.04	0.05
Ash (%)	99.78	99.74	99.34	99.34	99.13	97.40	95.16	98.67	98.23	98.69
Density (g/cc)	2.71	2.71	2.70	2.73	2.79	2.85	2.85	2.74	2.76	2.73

Run # Date	15 11/10/87	15 (DUP) 11/10/87	18 11/10/87	7 11/11/87	9 11/11/87	11 11/11/87	13 11/11/87	16 11/12/87	17 11/12/87	19 11/13/87
Moisture (%)	1.36	1.04	0.83	0.69	0.61	0.59	0.77	0.61	0.50	0.71
Carbon (%)	1.30	1.23	3.04	0.92	0.48	1.19	1.47	1.24	1.95	0.44
Hydrogen (%)	0.15	0.18	0.20	0.07	0.08	0.10	0.12	0.08	0.17	0.08
Oxygen (%)	1.92	2.39	6.56	0.93	0.51	1.31	2.35	2.05	2.67	0.54
Nitrogen (%)	0.03	0.02	0.07	<0.02	<0.02	0.04	0.06	0.07	0.07	<0.02
Sulfur (%)	0.04	0.04	<0.01	<0.01	0.04	0.05	<0.01	<0.01	0.04	<0.01
Chlorine (%)	0.04	0.05	0.05	0.05	0.03	0.05	0.05	0.05	0.06	0.04
Ash (%)	96.52	96.09	90.08	98.03	98.86	97.26	95.95	96.51	95.04	98.90
Density (g/cc)	2.87	2.84	2.85	2.75	2.74	2.97	2.87	2.86	2.85	2.75

ash and moisture. Each separate analysis, representing a different day or different run, defines a separate column in the table. Results are in units of percent (%, by weight).

Tables 6-37 and 6-38 also present the results of total chlorine, higher heating value (HHV) and as-received bulk density determinations by Huffman Laboratories. Total chlorine was determined by an ignition-and-trap method (ASTM Method D-2361 or for low chlorine levels, Method D-808). The higher heating value was determined using a bomb calorimeter procedure (ASTM Method D-2015-77). Density was determined using a 250 ml sample.

6.9 Other Liquids Characterizations

The scrubber make-up water and holding tank water were subjected to several other types of analyses.

Tables 6-39 and 6-40 present the results of total dissolved solids (TDS), total suspended solids (TSS), pH, chloride and total organic carbon (TOC) analyses on the scrubber make-up water and scrubber liquor, respectively. TDS and TSS concentrations were determined by filtering the liquid sample and then drying and weighing the filter for TSS, and drying and weighing the filtrate for TDS. pH was determined with a pH meter (EPA Method 150.1); chlorine concentration was measured with a ion chromatograph. TOC was determined by taking the difference of the total carbon (complete combustion to CO2) and the inorganic carbon (CO2 released by acid). Since the same water (from a large tank) was used throughout this study, it was not necessary to take a sample on each run. Enough samples (one per day) were taken to ensure that the water was well characterized and not subject to change. For the scrubber liquor, each separate analysis, representing a different day or different run, defines a separate column in the table.

6.10 Concentration of Soluble Chromium (Cr+6)

Table 6-41 presents the results of the soluble chromium analysis carried out on the combined impinger liquors and probe and nozzle washings of

TABLE 6-39. RESULTS OF ANALYSIS OF WATER QUALITY PARAMETERS ON THE SCRUBBER MAKE-UP

Parameter:		pH	TSS	TDS	Cl	TOC
Date	Time	pH	(mg/L)	(mg/L)	(mg/L)	(mg/L)
11/3/87	1930	8.27	0	344	26.9	< 1
11/4/87	2115	8.21	0	327	25.7	< 1
11/5/87	2000	8.31	0	306	24.8	< 1
11/6/87	1700	8.33	0.205	325	25.3	< 1
11/9/87	1045	7.46	0.84	332	---	< 1
11/10/87	1445	7.67	0	348	---	6
11/11/87	1015	7.48	0	347	---	4 a
11/12/87	1150	7.72	0	353	---	4 a
11/13/87	1130	8.02	0	366	---	2 a

a: Value less than 5 times the detection limit.

TABLE 6-40. RESULTS OF ANALYSIS OF WATER QUALITY PARAMETERS ON THE SCRUBBER LIQUOR

Parameter:		pH	TSS	TDS	Cl	TOC
Date	Time	pH	(mg/L)	(mg/L)	(mg/L)	(mg/L)
11/3/87	1930	8.65	177.8	2609	675.3	4 a
11/4/87	2040	7.25	81.9	2653	672.5	< 1
11/5/87	1845	7.67	63.4	2277	513.7	< 1
11/6/87	1900	8.57	557	2390	662.2	< 1
11/9/87	1045	7.56	119	787	---	6
11/9/87	1230	7.45	80.1	791	---	< 1
11/9/87	1700	7.90	67.6	1064	---	< 1
11/10/87	1030	7.73	172	1004	---	2 a
11/10/87	1230	6.87	78	1056	---	< 1
11/10/87	1445	6.19	89.8	1145	---	< 1
11/10/87	1630	7.79	129	914	---	4 a
11/11/87	1015	7.85	62.7	875	---	4 a
11/11/87	1230	7.91	69.9	955	---	3 a
11/11/87	1445	7.96	49.4	944	---	5
11/11/87	1645	7.97	70.8	1101	---	2 a
11/12/87	1145	7.46	96.3	750	---	4 a
11/12/87	1345	7.76	65.9	864	---	2 a
11/13/87	1130	8.19	59.6	722	---	6

a: Value less than 5 times the detection limit.

TABLE 6-41. SOLUBLE CHROME DETERMINATIONS IN THE FLUE GAS

Location	Primary	Primary	Primary	Primary	Secondary	Secondary	Secondary	Secondary
Date:	11/4/87	11/5/87	11/5/87 Duplicate	11/6/87	11/4/87	11/5/87	11/5/87	11/6/87 Duplicate
Time:	0941-1130	0902-1002	0902-1002	1427-1527	0938-1118	0851-1000	0851-1000	1429-1529
Detected Mass (ug):	<137	<142	<142	<145	<250	<206	<206	<247
Volume (m^3):	0.9098	0.5877	0.5877	0.6220	1.1644	0.8291	0.8291	0.9349
Concentration (ug/m^3):	<151	<242	<242	<233	<215	<248	<248	<264

the all-glass sampling train specially designed for chromium testing. In the field, the chromium was chelated with ammonium pyrrolidine dithiocarbamate and the complex extracted into MiBK (methyl isobutylketone). The MiBK was later analyzed for chromium on a specially adapted atomic absorption spectrophotometer. Samples were taken on both the primary and secondary off-gases during the SITE testing. Results are reported in units of ug/m^3.

6.11 Concentration of Vapor Phase Lead in the Primary Off-gases

Table 6-42a and b presents the results of the special lead sampling conducted on the primary furnace off-gases. A high temperature probe extracted a filtered sample of the primary gas and maintained it at 450°F all the way to the injection port of a flame atomic absorption spectrophotometer set up at the site. Testing extended over the first four days of the study, but very little or no lead was detected. Therefore, since the parametric studies of the second week were to be run at equal or lower primary chamber temperatures and shorter residence times making lead vaporization less likely, the sampling effort was curtailed to just the four days, and the results of the lead testing are reported in terms of mostly less-than-detection-limit values. A separate report describing the lead testing study, which was not set out in the QAPP, is attached.

6.12 Results of the Continuous Flue Gas Monitoring

During the continuous runs (second week), since the runs were short and interest was centered upon atmospheric emissions, the CEM system was not switched back and forth but left mostly on the secondary off-gas.

Table 6-43 presents hourly averages for each day of testing for the five flue gas components of interest: oxygen (O_2), carbon monoxide (CO), carbon dioxide (CO_2), total nitrogen oxides (NO_x), and total hydrocarbons (THCs). Samples of flue gas were extracted and delivered via heat-traced teflon tubing to a distribution manifold in the Radian sampling trailer. From the manifold, each of the five instruments pulled the required volume of gas

TABLE 6-42a. VAPOR PHASE LEAD CONCENTRATIONS IN THE PRIMARY FURNACE OFF-GAS THE SHIRCO INCINERATOR AT ROSE TOWNSHIP, MI

Date	Time	Run	ppbv Pb[a]
11-3-87	1528	1	<2.7
	1551	2	<2.7
	1613	3	<2.7
	1646	4	<2.7
11-4-87	1736	1	2.7
	1838	2	<2.7
	1934	3	<2.7
11-5-87	1035	1	<2.7
	1108	2	2.7
	1325	3	<2.7
	1442	4	<2.7
	1515	5	<2.7
	1547	6	<2.7
	1614	7	<2.7

[a] Vapor phase lead concentration with the liquid phase to vapor phase correction factor applied.

TABLE 6-42b. LEAD CONCENTRATION IN SAMPLE ACQUISITION TUBING RINSE AS CONCENTRATION IN GAS PHASE

Date	Liquid Phase Pb Concentration ug/mL	Rinse Volume mL	Sample Time minutes	Sample Flow LPM	ppbvPb[a]
11-3-87	0.14	64.8	150	6.705	<2.7
11-4-87	0.090	112.9	72	6.006	3.4
11-5-87	0.31	147.4	174	7.081	5.4

[a] Vapor phase lead concentration with the liquid phase to vapor phase correction factor applied.

TABLE 6-43. CONTINUOUS EMISSION MONITORS AVERAGED RESULTS

Date	Time	Location	O2 (%)	CO2 (%)	CO (ppm)	NOx (ppm)	NMHC (ppm)
11/03/87	10:30-10:45	Stack	9.2	7.9	2	129	6
11/03/87	10:50-11:10	Primary	15.5	4.4	7	118	7
11/03/87	11:15-12:00	Stack	8.3	9.0	3	128	5
11/03/87	12:00-13:00	Stack	8.4	8.9	3	127	2
11/03/87	13:05-13:15	Primary	15.9	4.2	4	101	3
11/03/87	13:20-13:25	Stack	8.6	8.7	3	136	8
11/03/87	13:35-13:40	Primary	15.5	2.9	1	60	4
11/03/87	13:45-14:00	Stack	8.3	8.7	2	133	4
11/03/87	14:00-15:00	Stack	8.9	8.5	3	130	3
11/03/87	15:05-15:25	Primary	17.1	3.4	3	92	2
11/03/87	15:30-16:00	Stack	9.8	7.8	2	121	3
11/03/87	16:00-16:25	Stack	9.8	7.9	2	122	8
11/03/87	16:30-16:45	Primary	15.5	4.4	3	95	4
11/03/87	17:20-17:35	Stack	8.9	7.7	2	124	6
11/03/87	17:40-17:55	Primary	16.3	4.6	2	99	4
11/03/87	18:00-18:45	Stack	9.5	8.1	2	133	7
11/04/87	10:35-11:10	Scrubber	7.8	8.4	1	118	1
11/04/87	11:10-11:40	Primary	15.4	5.1	3	110	6
11/04/87	11:40-12:45	Scrubber	8.2	8.6	2	117	5
11/04/87	12:45-13:00	Primary	16.3	4.2	5	92	5
11/04/87	13:00-14:00	Scrubber	7.3	7.9	1	115	4
11/04/87	14:00-15:00	Scrubber	7.7	8.8	2	116	4
11/04/87	15:00-15:25	Primary	14.2	6.2	5	114	5
11/04/87	15:25-16:00	Scrubber	7.9	8.2	2	113	6
11/04/87	16:00-17:00	Scrubber	7.6	7.4	2	109	6
11/04/87	17:00-17:10	Scrubber	9.0	7.8	3	120	4
11/04/87	17:10-17:25	Primary	15.5	4.5	5	108	4
11/04/87	17:25-18:00	Scrubber	8.1	7.1	1	112	3
11/04/87	18:00-19:00	Scrubber	8.3	7.0	1	107	2
11/05/87	09:00-10:00	Scrubber	8.2	8.5	0	98	--
11/05/87	10:00-11:00	Scrubber	7.9	6.6	0	108	--
11/05/87	11:00-12:00	Scrubber	8.0	6.5	0	106	--
11/05/87	12:00-12:10	Scrubber	11.8	7.9	0	96	--
11/05/87	12:10-12:20	Primary	17.0	4.1	4	97	--
11/05/87	12:20-13:00	Scrubber	10.1	8.2	0	104	--
11/05/87	13:00-14:00	Scrubber	9.9	7.9	0	101	--
11/05/87	14:00-15:00	Scrubber	10.4	5.8	0	74	--
11/05/87	15:00-16:00	Scrubber	7.9	9.6	1	127	0
11/05/87	16:00-17:05	Scrubber	7.9	9.8	1	125	2
11/05/87	10:05-17:20	Primary	15.6	6.4	2	123	2
11/05/87	17:20-18:00	Scrubber	8.2	9.4	0	127	3
11/05/87	18:00-18:30	Scrubber	8.2	9.9	1	126	3
11/05/87	18:30-18:40	Primary	14.4	7.6	3	120	2

+ All values given are on a dry volume basis

(Continued)

TABLE 6-43. (Continued)

Date	Time	Location	O2 (%)	CO2 (%)	CO (ppm)	NOx (ppm)	NMHC (ppm)
11/06/87	10:30-11:00	Secondary	6.9	9.9	0	140	3
11/06/87	11:00-12:00	Secondary	9.5	7.7	0	126	2
11/06/87	12:00-12:45	Secondary	6.6	9.9	1	135	2
11/06/87	12:45-13:20	Primary	16.9	4.0	6	109	4
11/06/87	13:20-14:00	Secondary	8.2	8.7	2	144	4
11/06/87	14:00-15:00	Secondary	8.1	9.0	2	142	3
11/06/87	15:00-15:50	Secondary	8.3	8.6	2	147	3
11/09/87	10:00-11:00	Secondary	10.6	7.3	0	96	3
11/09/87	11:00-12:00	Secondary	10.6	7.3	0	100	3
11/09/87	12:00-13:00	Secondary	13.5	5.1	0	82	3
11/09/87	13:00-14:00	Secondary	10.2	6.9	1	111	3
11/09/87	14:00-15:00	Secondary	7.3	9.0	1	133	4
11/09/87	15:00-16:00	Secondary	10.7	6.7	1	114	5
11/10/87	09:00-10:00	Secondary	9.2	8.3	0	85	2
11/10/87	10:00-11:00	Secondary	8.9	8.9	0	87	3
11/10/87	11:00-12:00	Secondary	8.6	9.0	1	82	3
11/10/87	12:00-13:00	Secondary	8.1	9.1	1	78	3
11/10/87	13:00-14:00	Secondary	7.5	9.4	1	72	3
11/10/87	14:00-15:00	Secondary	10.2	7.3	1	77	5
11/10/87	16:00-17:00	Secondary	10.7	6.9	2	79	3
11/11/87	09:00-10:00	Secondary	7.6	9.2	0	127	3
11/11/87	10:00-11:00	Secondary	16.5	3.1	1	65	3
11/11/87	11:00-12:00	Secondary	7.9	8.3	1	151	2
11/11/87	12:00-13:00	Secondary	8.2	8.1	2	148	3
11/11/87	13:00-14:00	Secondary	7.6	8.6	2	155	3
11/11/87	14:00-15:00	Secondary	7.9	8.3	1	153	3
11/11/87	15:00-16:00	Secondary	7.5	8.5	1	147	3
11/11/87	16:00-17:00	Secondary	13.8	4.6	1	85	3
11/12/87	10:00-11:00	Secondary	7.0	9.9	0	142	2
11/12/87	11:00-12:00	Secondary	9.3	8.3	0	117	2
11/12/87	12:00-13:00	Secondary	11.7	6.2	0	91	3
11/12/87	13:00-14:00	Secondary	11.4	6.3	1	90	2
11/13/87	09:30-10:00	Secondary	9.8	7.5	0	77	4
11/13/87	10:00-11:00	Secondary	8.0	9.3	0	70	3
11/13/87	11:00-12:00	Secondary	8.0	9.3	0	69	2

+ All values given are on a dry volume basis for analysis. Every fifteen minutes or so, the source of the sampled gas was alternated between the primary and secondary streams. Five minute averages for each of the analytes are available in the supplemental data appendix. All concentrations are given given in units of percent or ppmv.

6.13 Meteorological Monitoring

Table 6-44 presents the hourly averages of the wind direction and velocity. The location of the upwind and downwind hivol samplers is also indicated in the table for each day. Since the samplers were too close to the site (within 50 m) to truly sample representative air from all parts of it, exact measures of upwind/downwind percentages (i.e., wind roses) are not justified. Rather, these data should be used to simply indicate that for a period of time each day, the hivol samplers were generally upwind or downwind of the site handling and incineration activities.

TABLE 6-44. METEOROLOGICAL DATA FOR AMBIENT AIR SAMPLES

Date: Position:[a]	11/3/87 A		11/4/87 B		11/5/87 C		11/6/87 C	
Time	Wind Direction (degrees from north)	Wind Speed (mph)	Wind Direction (degrees from north)	Wind Speed (mph)	Wind Direction (degrees from north)	Wind Speed (mph)	Wind Direction (degrees from north)	Wind Speed (mph)
0800	—	—	240	4.0	300	5.9	—	—
0900	—	—	270	5.6	300	8.3	—	—
1000	240	4.3	270	5.6	300	8.3	300	5.9
1100	260	4.8	270	5.6	300	9.1	300	7.1
1200	260	4.8	270	5.6	300	8.3	300	7.7
1300	260	5.0	270	5.6	320	8.3	300	6.3
1400	240	5.6	270	5.9	300	8.3	300	6.3
1500	240	5.3	270	6.3	300	8.3	300	6.3
1600	240	4.5	270	6.3	300	7.7	300	5.6
1700	240	3.6	270	6.3	300	7.1	300	5.6
1800	210	4.0	270	5.9	300	6.3	—	—
1900	210	4.5	260	4.3	300	5.9	—	—
2000	—	—	270	5.0	300	5.6	—	—
Average	240	4.6	270	5.5	300	7.5	300	6.3

Date: Position:[a]	11/10/87 C		11/11/87 C		11/12/87 C		11/13/87 B	
Time	Wind Direction (degrees from north)	Wind Speed (mph)	Wind Direction (degrees from north)	Wind Speed (mph)	Wind Direction (degrees from north)	Wind Speed (mph)	Wind Direction (degrees from north)	Wind Speed (mph)
0800	—	—	330	1.7	—	—	240	2.0
0900	330	6.7	330	2.5	—	—	240	2.2
1000	330	6.7	320	3.3	270	2.0	270	2.9
1100	330	6.7	300	4.0	260	2.5	270	3.3
1200	330	6.7	280	3.7	240	2.5	270	3.3
1300	330	6.3	300	3.7	240	3.3	—	—
1400	330	5.9	300	3.7	240	3.3	—	—
1500	330	5.9	—	—	—	—	—	—
1600	330	6.3	—	—	—	—	—	—
1700	330	6.3	—	—	—	—	—	—
Average	330	6.4	310	3.2	250	2.7	260	2.7

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[a] Letters (A, B, C) refer to positions of sampler as indicated on Figure 3-1. Data from 11/9/87 not available due to station manfunction. Notes recorded that samplers were set at C position, wind direction at 300-330 degrees from north, and wind speed at 6-7 mph.

7.0 DISCUSSION OF RESULTS

The results reported in Section 6 represent all of the data collected during the trial burn at the Rose Township site. Readers interested in particular aspects of the test should use those data to reach their own conclusions. However, there are certain trends, patterns and conclusions contained within the numbers which may be of general interest to some who are not familiar with the data. In this section, the data are discussed (more or less on a table-by-table basis) and any unusual, unexpected or particularly interesting results are pointed out.

7.1 Discussion of Particulate Matter and Metals Results

The particulate stack emissions from the Shirco Incinerator at the Rose Township site were all below 0.03 gr/dscf corrected to 7% oxygen. The RCRA emission limit, applicable to most hazardous waste incinerators, is 0.08 gr/dscf. The removal rate of particulate material across the wet venturi (50 to 85 percent removal, see Table 6-2) improved on each of the first three days during the SITE testing; the stack emissions were noticeably less during the second week.

The particulate loading seemed to be somewhat influenced by certain operating parameters. During the second week for example, emissions were greater when the secondary combustion temperature was maintained at 2200°F than when maintained at 1800°F. Higher feed rates correlated with lower emissions, and longer residence times in the primary chamber usually resulted in higher particulate loadings. Other operational parameters, such as temperature in the primary chamber and the flow rate of the flue gas were not correlated with mass emissions.

The HCl burden in the flue gas was proportional to and nearly equal to the mass emission rate (lbs/hr). As expected, the wet venturi scrubber much more effectively removed the HCl than it did the particulate matter.

Whereas only 50 to 85 percent of the particles were removed, greater than 98 percent of the HCl was taken out of the flue gas through the venturi.

Because the contaminated soil samples were not homogeneous, the metals content of the feed material varied from sample to sample; see Table 6-3. Of particular interest, lead levels were high and variable during the first week of SITE testing, but were more regular during the second week and averaged about 600 ppm. Barium also averaged about 600 ppm. Chromium ranged from 59 to 180 ppm with a median of about 90 ppm. Nickel and copper were not too variable and generally between 15 and 34 ppm. Zinc was usually between 200 and 350 ppm. Other metals were generally at or below their detection limits.

Most of the metals were enriched in the ash over the feed material; see Table 6-5. This is to be expected since, as shown in the ultimate analysis (Tables 6-37 and 6-38), about 10 percent oxygen, 3 percent carbon and 12 percent moisture were lost during incineration. The combination of these loses would explain about a 33 percent enrichment. Ni, V and Zn increased approximately between 10 and 40 percent. Barium and lead increased in the ash, on the average by about 60 percent. An explanation for this result is in the uncertainty inherent in measurement of these two elements. Table 8-19 shows that replicate analysis of both soil and ash samples did not always yield the same results. This uncertainty is perhaps caused by matrix effects of the medium, since the variability is greater than normally experienced in lead and barium analyses. The daily ratio of lead in the ash to lead in the feed material was also very inconsistent during the first week when the lead levels in the feed varied widely. Copper and chromium concentrations held steady or decreased slightly after incineration. Most other metals remained below detection limits in the ash.

The ratio of metals in the flue gas particulate material were similar to those in the feed material. Ba, Cr and Ni were slightly enriched in the fly ash. The ratio of lead to total particulate matter was similar to that seen in the feed material.

7.2 Discussion of PCBs Results

The PCBs were analyzed by gas chromatography/mass spectroscopy. Each sample was spiked with BFB (4-bromofluorobenzene) and based on the response to that compound, combined with dilutions, sample size, known response factors for each compound, tailing factors, etc., a lower detection limit was calculated for each individual sample and each analyte. Also, if there were large amounts of one analyte in a particular sample, the sensitivity of the instrument to all other species had to be reduced so that the detector would not be saturated. Thus, the detection limits given in Tables 6-10 through 6-15 vary from compound to compound and from run to run. If one congener was high, other detection limits rose concomitantly.

The total amount of toxic PCBs in a particular sample is not simply the sum of all the PCBs reported. Since the mono- and di-chloro congeners are not nearly so toxic, EPA Regulations (40 CFR 761.3) suggest that the mono-congener be divided by 50 and the di-congener by 5 and the rest of the tri-through deca-congeners by summed directly. Also, if the concentration of a congener is below the detection limit, it is not to be included in the sum. Such a procedure was followed in this report to arrive at the total toxic PCB contribution in each sample as given in the tables.

The contaminated soil (feed material) always contained PCBs, but the concentrations showed a large range of variation (see Table 6-10). The tetrachlorobiphenyl congener was always the most prominent, and ranged from 8.8 ppm to 480 ppm. The higher concentrations were encountered during the second week of testing. The total toxic PCBs ranged from 10.2 ppm to 669 ppm.

Very little or no toxic PCBs were seen in the ash, unless the incinerator's primary chamber was operated at the 900°F condition. In the three runs where that was the case, small amounts survived the heating (2.08, 0.17 and 3.40 ppm). During the SITE operation, parts per billion quantities were detected in the ash (37, 112, and 3 ppb were the three totals). The highest other ash reading during the seventeen total runs was 77 ppb. The percentages of toxic PCBs removed in the soil-to-ash heating (i.e., the reductions in concentrations) for the SITE burns were 99.64%, 99.68%, and 99.99% for the first three days. For the 900°F primary temperature condition, the percentages of removal were 99.36%, 99.96% and 83.19%; the last number is low partially because the PCB concentration in the feed during that run was low (the second lowest during the seventeen runs).

Very little of the PCBs were detected in the flue gas stream. During the SITE burns, trace quantities were seen in the primary off-gas and secondary off-gas on only one of three runs (not the same run). The percentages of mass flow in the off-gas to mass flow in the feed material (equivalent to destruction efficiencies), were calculated as follows:

$$\frac{\text{mass in (feed material)} - \text{mass out (flue gas)}}{\text{mass in (feed material)}} \times 100$$

Results are given in Table 7-1. Where no PCBs were detected, all of the "less than" values were summed to produce a worst case scenario. No PCBs were detected in the scrubber liquors (Table 6-13). It appears in Table 7-1 that there are more PCBs in the secondary off-gas than the primary. However, PCBs were detected only in one sample (11/5, secondary) and the other removal efficiencies simply reflect the detection limits, which were somewhat lower in the primary than the secondary.

Small amounts of PCBs were seen on the ambient air filters, including some upwind filters. However on three days when the PCBs in the feed material were highest (see Table 6-10), 11/9, 11/10, 11/11, there were increases of PCBs detected downwind from the site. See Table 7-2.

TABLE 7-1. PERCENTAGE OF PCBs REMOVED BY INCINERATION

Date	Primary Chamber %	Secondary Chamber %
11/3	99.9999	>99.9925
11/4	>99.9997	>99.9977
11/5	>99.9991	99.9982

TABLE 7-2. TOTAL PCBs DETECTED ON AMBIENT FILTERS

Date	PCBs Upwind (ng/m3)	PCBs Downwind (ng/m3)
11/3/87	14.9	2.97
11/4/87	3.55	3.25
11/5/87	--	--
11/6/87	15.4	10.1
11/9/87	15.8	21.8
11/10/87	8.4	48.7
11/11/87	5.4	12.8
11/12/87	--	0.4
11/13/87	--	--

7.3 Discussion of Dioxins and Furans

Very small amounts of tetrachlorodibenzofuran were detected in a about half of the soil samples. However, concentrations were always at or below 0.1 ppb (the detection limit was 0.044 ppb). No other PCDDs or PCDFs were detected. No dioxins or furans were detected in the flue gas where the detection limit for the tetrachlorodibenzofuran was about 0.2 ng/m3 in the primary off-gas and about 1.0 ng/m3 in the secondary off-gas. Nor were any dioxins or furans detected in the scrubber make-up water or scrubber liquor. Two samples of ash contained small amounts of tetrachlorodibenzofuran; the 0.3 ppb concentration in the ash of 11/12 was the highest level found anywhere in this study; see Table 6-20.

7.4 Discussion of the Semi-Volatile Organic Compounds

In all of the feed material, scrubber make-up, scrubber liquor and ash samples, only one semi-volatile organic compound on one day was detected -- bis(2-ethylhexyl)phthalate (feed material on 11/5); see Table 6-21.

In the off-gases from the primary chamber, five species were detected at low levels during two days of testing. All five were oxygenated benzene rings: three species of phthalates, benzoic acid and p-chloro-m-cresol. Concentrations were all small and less than five times the detection limit. It also should be noted that phthalates often contaminate laboratory samples (they are found in most plastics), and two did show up in the filter blanks (see Table 8-23).

Similar results occurred in the secondary off-gases. The same three phthalates were detected as was benzoic acid. Concentrations were similar to those seen in the primary off-gas. Benzoic acid and di-n-butyl phthalate were detected in all three runs sampled. Thus, the semi-volatile burden in the off-gases was very small, and part of what was detected was probably due to contamination.

7.5 Discussion of Volatile Organic Compounds

The feed material, when analyzed for volatile organic compounds by Method 8240, contained virtually nothing. There was some methyl ethyl ketone and trichloromethane detected, but both of these compounds were also detected at about the same levels in the solvent blank; see Table 6-26. The furnace ash analyses was similar except for one rather high methylene chloride peak; see Table 6-30. This unusual reading for methylene chloride on the 11/4 sample is probably due to some sort of contamination; methylene chloride was used extensively as a solvent during the testing.

The scrubber make-up water was delivered from a tank truck. When a barrel was filled with it, a thin oily film was noticeable on the surface. The driver was not sure about what had been in the truck before the water; some type of oil was likely since there were extensive oil stains on the outside of the tank. The volatile analysis of the make-up water and the scrubber water consistently detected small amounts of benzene and toluene, but very little else; see Tables 6-28 and 6-29.

There was a richer variety of volatile organic compounds in the flue gas. Apparently, the volatile compounds were not released from the soil until heating; also, some might have been created from larger organic species by the pyrolysis. The following discussion refers to the six page Table 6-27, the VOST results.

In the primary off-gas, halomethanes (Cl, Br, I) were usually present and in non-trivial amounts, sometimes over 100 ug/m3. There were also several other chlorinated species frequently detected (e.g., chlorobenzene, methylene chloride, trichlorofluoromethane). Certain hydrocarbons were usually found (e.g., toluene, xylenes, styrene, benzene, ethylbenzene), some at levels also well above 100 ug/m3. Some oxygenated hydrocarbons, perhaps the partial products of combustion, were detected (e.g., acetone and fairly large amounts of acrolein). Finally a few other compounds sometimes were detected in the primary off-gases, notable among them was carbon disulfide on 11/3. The total organic burden was substantially higher on 11/3 than on 11/4 or 11/5.

The concentrations of most organic species were reduced in the secondary off-gases, but the ratios were very inconsistent. Concentrations varied substantially from one VOST tube to another (each was a twenty minute sample) implying fluctuating concentrations. For example, on 11/5, toluene in four VOST tubes in the primary off-gas measured 4.1, 31.4, ND, 4.8 ug/m3, for an average of about 13 ug/m3; in the secondary off-gas during the same run, the toluene concentrations were 0.6 and 0.9 ug/m3. Thus, the toluene was obviously reduced substantially. Less apparent would be the case of the xylenes on 11/4. In the primary off-gas, they measured at 1.2, ND, ND ug/m3; in the secondary for that run they measured at 1.38, 0.4, 0.4 ug/m3. What happened in this case is less clear. And then there were cases where products may have been generated during secondary combustion. A case in point is again carbon disulfide. On 11/3, carbon disulfide was measured in the primary off-gas at 14.3, 0.9, 10.7 ug/m3. In the secondary off-gas the results were 1.38 and ND ug/m3. But on the next day, carbon disulfide in the primary was ND, ND, ND, while in the secondary it was measured at 14.9, 9.4, 8.5 ug/m3. In conclusion, a general trend cannot be defined, and each organic compound must be looked at individually. For the most part, concentrations were reduced across the secondary combustion chamber, and the variety of species decreased, but not enormously. In a few cases, some new products appeared to be created.

7.6 Discussion of EP Toxicity Leachate Metals Analysis

In the EP Toxicity leaching procedure (Method 1310), the metals were leached out of the soil and ash samples and the leachate analyzed for eight specific metals; see Table 6-31 and 6-33. Seldom were any Ag, Hg or Se detected. Small amounts of Cd (about 0.01 mg/L) and As (0.003 mg/L) were seen in the ash. Chromium was often about 0.05 mg/L in the feed material, and averaged nearly the same in the ash samples, although in the ash chromium was more variable with higher numbers seen during he first week. Barium was often near 0.9 mg/L in the feed material and also more variable in the ash samples, but of the same general concentration. Of greatest interest was lead, and it was often found at about 0.2 mg/L (range 0.05 to 0.67 mg/L) in the feed material. As with chromium and barium, the analyses showed much greater variability in the ash samples, where the lead ranged from ND to 4.1 mg/L (near the regulatory limit of 5.0 mg/L). No obvious correlation exists between in and out samples. For example, on the run where the highest level of lead was measured in the ash, 4.1 mg/L (on 11/9, run #14), the lead in the feed material for that run was the smallest seen during any run, 0.05 mg/L. Other than the one high lead value at 80 percent of the regulatory level, no other metal concentrations were close to the statutory limit.

The EP Toxicity leachate of the scrubber liquors contained almost no lead (detected only twice in 16 samples). Chromium was higher than in the feed material or ash ranging from 0.055 to 0.52 mg/L. As, Ba, Cd and Se were also consistently detected.

7.7 Discussion of TCLP Leachates for Metals and Semi-Volatile Organics

The TCLP leachates of solid samples were analyzed for both metals and semi-volatiles; see Tables 6-34 and 6-36. The liquids were analyzed only for metals; see Table 6-35. Table 6-29 presents the results of volatile organic analysis of scrubber liquors. Again, As, Se and Hg were almost never detected. In the feed material, barium was consistently detected at about 2.0 mg/L, about double that found with the EP Toxicity procedure. Lead was also measured at higher levels than with the EP Toxicity procedure. In the feed material, lead ranged from 0.44 to 7.0 mg/L, but most values were between 0.5 and 1.0 mg/L. The chromium levels in the feed were about ten times those found with the EP Toxicity procedure. Still, only lead was near its regulatory limit, and even lead, except for the unusually high level on one run on 11/12, was always less than 20 percent of the statutory level. Lead and chromium levels in the ash were similar to those found in the feed material, while barium was often only half its concentration level in the feed.

In the leachate of the scrubber liquors, again almost no lead was found, and most other results were very similar to those found with the EP Toxicity procedure.

The organic content of the TCLP leachate of the feed material contained almost nothing until the material collected after noon on 11/10. Rather suddenly, almost all of the organic species of interest in the procedure were detected, albeit in rather small quantities. This gives evidence to the inhomogeneity of the feed material. Almost no species were within twenty percent of the regulatory limit (nearest were chloroform, carbon tetrachloride, tetrachloroethylene, trichloroethene, and vinyl chloride). These species were not found in greater concentrations than many other compounds, but their regulatory limits are lower. After 11/10, some of the organic species were detected in a more or less hit-and-miss pattern (see Table 6-34), again all at relatively low levels. TCLP leachates of the ash samples contained only trace amounts of a few organics, most often benzene and toluene at levels usually below 0.01 mg/L.

7.8 Discussion of Ultimate Characterization of Solid Materials

The feed material contained approximately 11% water, 12% oxygen, 3% carbon, 1.5% hydrogen and about 0.1% chlorine; see Table 6-37. These numbers are consistent with the reported ash content (of the dry material) of about 83 percent. The higher heating value (HHV) and carbon content were consistent with other data from this study (e.g., PCB data, volatile and TCLP analyses) indicating there was a higher concentration of organic material on 11/10, levels more than double the average on the other days.

The ash material differed from the feed material in predictable ways; see Table 6-38. Carbon was reduced to well below 1 percent except when the primary chamber was run at 1200°F or below. At these lower temperatures the carbon was slightly over 1 percent except on run # 18 where the carbon in the ash was still above 3 percent. Nearly all of the chlorine was gone (below 0.05%). The ash content of all samples exposed to temperatures above 1200°F was always greater than 98 percent.

7.9 Discussion of Other Data

The analysis of the scrubber liquors, Table 6-40, revealed no unexpected results. There were occasionally high TSS levels, probably a result of were detected, albeit in rather small quantities. This gives evidence to the inhomogeneity of the feed material. Almost no species were within twenty percent of the regulatory limit (nearest were chloroform, carbon tetrachloride, tetrachloroethylene, trichloroethene, and vinyl chloride). These species were not found in greater concentrations than many other compounds, but their regulatory limits are lower. After 11/10, some of the organic species were detected in a more or less hit-and-miss pattern (see Table 6-34), again all at relatively low levels. TCLP leachates of the ash samples contained only trace amounts of a few organics, most often benzene and toluene at levels usually below 0.01 mg/L.

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7.9 Discussion of Other Data

The analysis of the scrubber liquors, Table 6-40, revealed no unexpected results. There were occasionally high TSS levels, probably a result of the sampling variability or, more likely, turbulence immediately prior to sampling. The liquors were always slightly basic. The TDS levels were rather predictable from run to run, the concentrations more or less reflected the total time of the run.

The special sampling for vapor phase lead and soluble chromium both were unable to detect any of either metal. In these cases, the detection limits represent some useful information; see Tables 6-41 and 6-42.

The continuous emission monitoring (CEM) data also are consonant with expectations. The oxygen was between 14 and 17 percent in the primary off-gases and generally between 5 and 11 percent in the secondary off-gases. Carbon dioxide levels followed the opposite trend, averaging about 5 percent in the primary and 9 percent in the secondary off-gases. Very little carbon monoxide or non-methane hydrocarbons were detected at any time. The NOx concentrations were generally about 120 ppm if the secondary combustion chamber was operated at 2200°F, and nearer 80 ppm when operated at 1800°F.

8.0 QUALITY ASSURANCE AND QUALITY CONTROL

The objective of quality assurance and quality control (QA/QC) efforts associated with sampling and analytical activities was to ensure that data collected are of known and sufficient quality to qualitatively and quantitatively characterize the sources of interest. Two primary aspects of the overall QA/QC effort are related to achievement of this objective. First, the QA/QC program forms a framework for controlling data quality within preestablished limits during execution of the sampling and analytical efforts. Second, the QA/QC program forms the basis for estimating uncertainty in the measurement data by providing information necessary for identifying and defining qualitative and quantitative limitations associated with these data. The following sections present a brief description of some of the key elements of the QA/QC effort and an assessment of measurement data quality based on QA/QC results.

Summary

Overall, QA/QC data associated with the Rose Township infrared incineration study indicate that the measurement data are acceptable and defensible. Samples were analyzed for PCBs, dioxins and furans, volatiles, semivolatiles, metals, TCLP extracts (volatiles, semivolatiles, and metals), EP extract metals, chloride in filters, hexavalent chromium, total organic carbon, and solids characterization. The most significant problems noted, from a QA/QC standpoint, were that the analyses for PCBs and semivolatiles were not carried out within the holding time specified by the QAPP. Selenium was not recovered in the matrix spike samples for solids, TCLP and EP extracts, and filters.

Analyses of solid, liquid, and filter samples for PCBs met most of the specifications in the QAPP, except for holding time. Blank recoveries were acceptable for all matrices. Surrogate spike recoveries tended to be low for dirt, higher weight PCBs in liquids, and ambient air filters. Recoveries were generally low for matrix spike/matrix spike duplicate compounds for ash, make up water and the filter blank sample. Repeatability of the matrix spike/matrix spike duplicate samples was excellent except for one sample of make up water. This sample was one of two of this type of matrix analyzed. Recovery for the other sample was within specifications.

Low recoveries for the PCB surrogate and matrix spike compounds suggest that reported results for field samples are probably lower than the true concentrations.

Analyses of dioxins and furans in solid and liquid samples met most of the acceptance criteria specified in the QAPP. Blank sample analyses showed no contamination from analytical sources. Although surrogate recoveries were low for the higher molecular weight dioxins and furans for both types of matrices, none of the native dioxins and furans were detected in field samples. Surrogates were recovered at about 30%, which suggests that actual field sample concentrations are probably no greater than about three times the laboratory stated detection limit. Repeatability for the compounds reported in two pairs of duplicates was excellent.

Quality control data for volatiles in solids, liquids, VOST samples, and TCLP extracts showed no significant problems in these analyses. Some common laboratory contaminants (methyl ethyl ketone, 1,2-dibromo-3-chloro propane, and xylenes) were detected in more than one blank analyzed with the solid samples. None of these compounds should be considered a significant contaminant since they were detected at less than five times the method detection limit. Recoveries of surrogate spike and matrix spike compounds was excellent for all four matrix types.

No quality control problems were detected with the semivolatile analyses except for the holding time violation. Surrogate and matrix spike recoveries are acceptable for almost all compounds. Surrogate recovery was low for 2-fluorophenyl in TCLP extracts of dirt and ash samples. This may have been due to exceeded holding time, however, recovery of the other five surrogate compounds was acceptable.

Analyses of metals were performed on solids, liquids, TCLP and EP extracts, and filter samples. Several elements (arsenic, barium, beryllium, cadmium, chromium, copper, lead, silver, vanadium, and zinc) were detected in the blanks above the detection limit. However, only one element (barium), in one blank sample, was recovered at greater than five times the detection limit. Therefore, analytical contamination should not be considered to be a major factor in the reported recoveries for field samples. Matrix spike recovery was acceptable for all elements except silver in filter samples, and selenium in all matrices but liquids. Precision of analyses measured by matrix spike duplicate analyses and analytical duplicate analyses was good. As expected, precision of analyses of solid samples was not as good as those for other matrices.

Silver was not recovered in matrix spikes of filter samples. A footnote on the laboratory report states that silver in the spike material is incompatible with the digestion process. Any silver originally present in the field samples might also be precipitated out in the digestion, and therefore, not detected.

Recovery for selenium was poor in all matrices except liquids. Recovery for three of four samples (0%, 0%, and 1%) averaged 0.33 percent. Selenium was not recovered in matrix spikes of TCLP or EP extracts, or filter samples. This implies that selenium could have been present and not detected in the field samples (i.e., false negative results).

Review of the results for chloride analyses in filters, hexavalent chromium, total organic carbon, and solids characterization shows good quality control. No problems were detected in the data.

Approach

The overall QA/QC program for the study was designed to fulfill two related purposes. First, it provided an organized framework for the sampling and analytical efforts. The program was designed to control data quality within preestablished limits and to ensure that it was adequate to achieve the objectives of the program. This involved first establishing data quality objectives for the parameters of interest. Next, protocols were defined for the critical aspects of the measurement effort, including:

- Sample collection, preservation, and storage;
- Calibration of instrumentation and apparatus;
- Data reduction, validation, and reporting;
- Documentation and sample custody; and
- Analytical quality control.

These protocols were tailored such that careful adherence to the prescribed protocols would, in most cases, result in achievement of both sets of objectives.

The second purpose of a QA/QC program is to provide the mechanism for data quality assessment. Quality control data provide information for identifying and defining qualitative and quantitative limitations associated with the measurement data. The following key types of QA/QC procedures provide the primary basis for quantitatively evaluating data quality:

- Field and laboratory blank samples;
- Spiked samples;
- Duplicate analyses; and
- Quality control check samples;

Blank samples play an especially important role in trace analysis efforts. They provide qualitative assurance that analytes detected in the samples are actually characteristic of the media sampled and are not artifacts of the sampling and/or analytical process. Results are presented in the following sections for analysis of laboratory reagent blanks, field blanks, and extraction blanks.

Laboratory reagent blanks address only the analytical process. Typically included with each batch of samples analyzed, they provide an ongoing check of the analytical system for systematic sample contamination. When evidence of contamination is indicated by blank values above preestablished levels, corrective action is initiated to identify and eliminate the source of contamination. If possible, the affected samples may be reanalyzed.

Field blanks reflect the combined effects of sample collection, handling, transportation, storage, and analysis. Since it is often not possible to resample when field blanks indicate possible sample contamination, field blank data are more often used to define qualitative limitations of the associated measurement data. The presence of analytes of interest in either the field or laboratory blanks suggests that corresponding field samples may have been similarly contaminated and that results for these analytes should be considered suspect. If the blank data show a given analyte at widely varying concentrations, or at concentrations comparable to those for field samples, then the field sample results should be viewed as possible false positives for that analyte. If, on the other hand, blank data indicate a given analyte at concentrations much lower than typical sample concentrations, then the sample data may usually be viewed as qualitatively reliable, but as having greater than normal quantitative uncertainty.

TCLP and EP extraction blanks provide information related to contamination resulting from the TCLP process. Analytes found in TCLP blanks should be considered when interpreting results for TCLP extracts of field samples.

Two special types of spiked samples are used as part of the analytical protocol for analysis of organic compounds by gas chromatography. These are matrix spiked samples and surrogate spiked samples. Matrix spiked samples are field samples to which known amounts of the analytes of interest have been added. Typically, both a spiked and an unspiked aliquot of the sample are analyzed. The difference in results for the two analyses are calculated and compared to the amount of spike added. Since actual samples are used for the determination, any matrix effects are taken into consideration. Usually expressed as a percentage of the spike amount, spike recovery provides a measure of the accuracy of the analysis. For a single sample, this includes the combined effects of bias, or systematic error, and variability due to imprecision. Averaging spike recoveries for multiple samples tends to "average out" the random error due to imprecision and provides an estimate of analytical bias.

Surrogate spiked samples are similar to normal spiked samples except that an unspiked aliquot is not analyzed. Instead, samples are spiked with a mixture of surrogate compounds that are chemically similar to the species of interest but are not expected to be present in actual field samples. Recovery of these surrogate compounds provides an estimate of the accuracy of the method during that single analysis.

Duplicate samples and duplicate analyses are used as indicators of measurement data precision. Precision is a measure of mutual agreement among individual measurements of the same property made under prescribed similar conditions. Variability among the measurements is attributable to random error due to imprecision. In the case of duplicate analyses, the analytical process is replicated for separate aliquots of a single sample with prescribed elements of the process held constant. For example, duplicate analyses are usually performed on the same day, by the same analyst, using the same instrument and the same calibration. Differences in results for duplicate analyses are attributable to random variability in the analytical process and are a measure of analytical precision.

Duplicate samples provide another measure of precision. The analysis of duplicate samples involves replicating sample collection (and associated sample handling activities) as well as analysis, precision estimates based on duplicate sample results incorporate imprecision due to sampling variability and analytical variability.

Both duplicate sample and duplicate analysis results provide data for precision estimates. Generally, however, duplicate sample data are used in a different way than results for duplicate analyses. Since analytical precision is primarily a function of the analytical procedures used, results for duplicate analyses may be used as an ongoing quality control check. Corrective action can be initiated when results indicate that analytical precision is not within acceptable limits. Results for duplicate samples, on the other hand, are more often used as a data quality assessment tool. Because of the lag between sample collection and availability of analytical results, it is usually not possible to initiate corrective action based on duplicate sample data. Also, variability in duplicate sample results typically includes a component of variability attributable to inherent nonhomogeneity of the sample matrix. Duplicate data of both types are useful as indicators of the degree to which results may be expected to vary by chance alone. This information is important whenever comparisons are made between measured values. Without it, it is difficult to know when to attribute observed differences to random measurement error and when to attribute them to acute differences.

For organic analyses, it is often the case that detectable levels of target analytes are not found in duplicate sample pairs, which precludes developing estimates of measurement variability. In this report, matrix spike duplicate results are used to assess repeatability. This is expressed as the relative percent difference (RPD) between duplicate spiked sample recoveries. The range of surrogate spike recoveries for a given matrix also provide a measure of analytical precision, expressed in terms of the standard deviation of percent recovery.

Quality control check samples (QCCS) are used to assess analytical performance under a given set of standard conditions. These are synthetic samples containing some or all of the analytes of interest at known concentrations and prepared independently from calibration standards. Typically analyzed with each set of analyses, QCCS results are often used as a real-time check of analytical system performance by comparing measured values to pre-established acceptability limits. Quality control check sample results may also be used to estimate analytical bias and accuracy by comparing measured results to theoretical concentrations. Although they do not address matrix effects like results for spiked samples, they do allow day-to-day variability to be considered and are useful in identifying trends.

8.1 QA/QC Results for PCB Analyses

The QA/QC efforts associated with PCB analyses included daily calibration verification, blank sample analyses, duplicate analyses, and matrix and surrogate spike analyses. Following initial multipoint calibration, the system was checked daily for continuing calibration. As part of the daily calibration verification, a single point calibration standard was analyzed, with agreement within 15 percent of the multipoint response required for analyses to proceed. The samples for PCB analyses were extracted within the 14 day holding time, but were not analyzed within the 40 day holding time.

8.1.1 Blank Sample Analyses for Solid Samples

Analyses of laboratory blank samples are presented in Table 8-1. Three PCBs were detected in one reagent blank. They are listed below:

- Trichlorobiphenyl (140 ng/L):
- Tetrachlorobiphenyl (920 ng/L); and
- Pentachlorobiphenyl (50 ng/L).

TABLE 8-1. BLANK DATA REPORTED FOR SOLID SAMPLES ANALYZED FOR PCBs

Compound	Reagent Blank (ng/L)	Reagent Blank (ng/L)	Reagent Blank (ng/L)	Reagent Blank (ng/L)
Monochlorobiphenyls	<20	<20	<20	<20
Dichlorobiphenyls	<20	<20	<20	<20
Trichlorobiphenyls	<50	140	<30	<30
Tetrachlorobiphenyls	<70	920	<50	<50
Pentachlorobiphenyls	<50	50	<30	<40
Hexachlorobiphenyls	<100	<90	<60	<70
Heptachlorobiphenyls	<100	<100	<90	<100
Octachlorobiphenyls	<100	<200	<100	<100
Nonachlorobiphenyls	<200	<200	<100	<200
Decachlorobiphenyls	<200	<200	<100	<200

There is evidence of contamination of laboratory glassware with PCBs resulting from the extraction process. Field sample data containing similar levels of tri-, tetra-, and penta-chlorobiphenyls should be viewed with care.

8.1.2 Spiked Sample Analyses for Solid Samples

Surrogate spike recoveries are summarized in Table 8-2. Four ^{13}carbon-labeled chlorinated biphenyls were added to samples to check recoveries in individual samples. These results are generally within expected limits, with some exceptions. Some surrogate recoveries were precluded due to sample dilution. These recoveries are denoted in the table by a "NR". Of the eight dirt samples in which surrogates were recovered, those with results outside the 50-125% recovery are listed below:

- Decachlorobiphenyl (48%, 47%, 39%, 46%, and 46%);
- Tetrachlorobiphenyl (45%, 32%, 47%, 47%, and 19%); and
- Monochlorobiphenyl (162%).

Surrogate recovery for Sample 1112 11:45 was outside the acceptance criteria on three of four surrogates. Of the 21 ash samples, all surrogates were recovered. Only decachlorobiphenyl (40% and 48%) was recovered outside the acceptance criteria (50-125%) specified by the method. Results for ash field samples may be biased low, since the average spike recovery was 65%.

Matrix spike/matrix spike duplicate results are summarized in Table 8-3 for solid samples analyzed for PCBs. These results provide a measure of accuracy (mean percent recovery) and precision (relative percent difference). The objectives for MS/MSD results were 50-125 percent recovery and 40 percent RPD. For sample 1109 11:00, the recoveries for dichlorobiphenyl (35%), tetrachlorobiphenyl (46%), and pentachlorobiphenyl (33%) were outside the accuracy objective, but were within the precision objective. Recoveries outside the 50-125 percent objective reflect interferences in the sample matrices which may affect analyte quantitation.

TABLE 8-2. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR SOLID SAMPLES ANALYZED FOR PCBs

Sample I. D.	Monochlorobiphenyls (% Recovery)	Tetrachlorobiphenyls (% Recovery)	Octachlorobiphenyls (% Recovery)	Decachlorobiphenyls (% Recovery)
Dirt				
1103	68	45[a]	58	52
1104	52	32[a]	52	39[a]
1105	76	47[a]	78	46[a]
1109 11:00	NR	NR	NR	NR
1109 12:45	80	47[a]	63	46[a]
1109 17:00	90	54	75	75
1110 10:45	62	59	57	47[a]
1110 12:45	NR	NR	NR	NR
1110 14:50	NR	NR	NR	NR
1110 16:45	NR	NR	NR	NR
1111 10:30	NR	NR	NR	NR
1111 12:45	NR	NR	NR	NR
1111 14:50	NR	NR	NR	NR
1111 17:00	NR	NR	NR	NR
1112 11:45	162[a,b]	19[a]	72	48[a]
1112 13:45	72	52	74	66
1113 12:30	NR	NR	NR	NR
Mean	71	44	66	52
Standard Deviation	11	12	9	11

(Continued)

TABLE 8-2. (Continued)

Sample I. D.	Monochlorobiphenyls (% Recovery)	Tetrachlorobiphenyls (% Recovery)	Octachlorobiphenyls (% Recovery)	Decachlorobiphenyls (% Recovery)
Ash				
1103	68	83	82	65
1104	57	70	76	55
1105	68	80	82	61
1105 ms	66	76	80	61
1105 ms	66	76	78	64
1109 11:00	103	79	75	76
1109 11:00 ms	118	86	81	92
1109 11:00 ms	101	81	76	88
1109 12:45	87	68	66	68
1109 17:00	99	82	78	83
1110 10:45	62	85	82	57
1110 12:45	51	83	82	64
1110 14:50	47	64	61	52
1110 16:45	53	54	52	40[a]
1111 10:30	51	78	77	54
1111 12:45	58	82	77	60
1111 14:50	67	88	88	62
1111 17:00	71	62	58	52
1112 11:45	80	66	63	55
1112 13:45	74	60	59	48[a]
1113 12:30	88	72	70	53
Mean	73	75	73	62
Standard Deviation	19	9	10	13

(Continued)

TABLE 8-2. (Continued)

Sample I. D.	Monochlorobiphenyls (% Recovery)	Tetrachlorobiphenyls (% Recovery)	Octachlorobiphenyls (% Recovery)	Decachlorobiphenyls (% Recovery)
Reagent blank	59	78	78	59
Reagent blank	100	86	80	84
Reagent blank	122	86	81	81
Reagent blank	102	84	79	82
Mean	95	84	80	77
Standard Deviation	23	3	1	10

[a] Outside accuracy objective (50-125% recovery) as specified by the method.
[b] Not used in mean or standard deviation calculations.
NR = Not Recovered.

TABLE 8-3. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR SOLID SAMPLES ANALYZED FOR PCBs

Compound	1105 Matrix Spike (%)	1105 Matrix Spike Duplicate (%)	RPD[a] (%)	1109 1100 Matrix Spike (%)	1109 1100 Matrix Spike Duplicate (%)	RPD[a] (%)
Ash						
Monochlorobiphenyls	81	81	0	55	51	8
Dichlorobiphenyls	55	54	2	35[b]	35[b]	0
Trichlorobiphenyls	89	87	2	51	52	2
Tetrachlorobiphenyls	76	76	0	46[b]	48[b]	4
Pentachlorobiphenyls	54	53	2	33[b]	33[b]	0
Hexachlorobiphenyls	84	85	1	57	58	2
Heptachlorobiphenyls	116	113	3	82	83	1
Octachlorobiphenyls	103	99	4	76	78	3
Decachlorobiphenyls	89	90	1	60	60	0

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$

[b] Outside accuracy objective (50-125% recovery) specified by the laboratory.

8.1.3 Blank Sample Analysis for Liquid Samples

Blank results for PCB analysis of liquid samples is presented in Table 8-4. Two compounds were detected in the blank above the detection limit:

- Trichlorobiphenyl (98, 62, 92, and 42 ng/L); and
- Tetrachlorobiphenyl (280, 400 and 360 ng/L).

There is some evidence of laboratory contamination. Field samples of liquids with values close to these limits may be regarded with suspicion.

8.1.4 Spiked Sample Analyses for Liquid Samples

Surrogate recoveries for liquid samples are presented in Table 8-5. Surrogate recoveries were below the accuracy objective (50-125%) specified by the method for all four surrogate spike compounds for two samples (1105 and 1112 Make Up Water). Recoveries outside the accuracy objective are presented below:

- Decachlorobiphenyl (40%, 45%, 44%, 43%, 33%, 47%, 48%, 44%, 44%, 28%, 46%, 46%, 43%, 38%, 49% and 48%);

- Octachlorobiphenyl (46%, 36%, 47% and 47%);

- Tetrachlorobiphenyl (43%, 41% and 43%) and

- Monochlorobiphenyl (43%, 44%, 46%, 34%, 32% and 38%).

Recoveries outside the 50-125 percent relative objective interferences in the sample matrices which affect analyte quantitation. Field sample results may be biased low.

TABLE 8-4. BLANK DATA REPORTED FOR LIQUID SAMPLES ANALYZED FOR PCBs

Compound	Reagent Blank (ng/L)	System Blank (ng/L)	Reagent Blank (ng/L)	Reagent Blank (ng/L)	Reagent Blank (ng/L)	Reagent Blank (ng/L)	System Blank (ng/L)	Reagent Blank (ng/L)	Reagent Blank (ng/L)
Monochlorobiphenyls	<20	<20	<30	<8	<10	<20	<20	<20	<30
Dichlorobiphenyls	<20	<20	<30	<9	<10	<20	<20	<20	<30
Trichlorobiphenyls	<40	98	<40	62	92	<40	<30	42	<60
Tetrachlorobiphenyls	<60	360	<90	280	400	<70	<50	230	<100
Pentachlorobiphenyls	<40	<40	<70	<20	<30	<50	<40	<40	<70
Hexachlorobiphenyls	<80	<80	<100	<40	<60	<100	<70	<70	<100
Heptachlorobiphenyls	<100	<100	<200	<50	<80	<100	<100	<100	<200
Octachlorobiphenyls	<100	<100	<200	<50	<70	<100	<100	<90	<200
Nonachlorobiphenyls	<100	<200	<200	<70	<100	<200	<200	<100	<200
Decachlorobiphenyls	<100	<200	<200	<70	<100	<200	<100	<100	<200

TABLE 8-5. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR LIQUID SAMPLES ANALYZED FOR PCBs

Sample I. D.	Monochlorobiphenyls (% Recovery)	Tetrachlorobiphenyls (% Recovery)	Octachlorobiphenyls (% Recovery)	Decachlorobiphenyls (% Recovery)
Scrubber Effluent				
1103	43[a]	66	73	40[a]
1103 ms	57	74	82	45[a]
1103 ms	44[a]	64	60	44[a]
1104	52	66	62	43[a]
1105	59	82	85	56
1109 10:45	60	43[a]	46[a]	33[a]
1109 11:00	61	82	79	58
1109 17:00	86	73	67	70
1110 10:30	62	59	57	47[a]
1111 10:15	56	54	55	48[a]
1111 12:30	91	82	75	73
1111 14:45	57	74	82	74
1111 16:45	61	76	80	74
1112 11:45	88	74	70	67
1112 11:45 ms	88	74	68	82
1112 11:45 ms	58	76	84	92
1112 13:45	78	76	73	67
1113 11:30	66	71	69	62
Mean	65	64	70	60
Standard Deviation	15	20	11	16

(Continued)

TABLE 8-5. (Continued)

Sample I. D.	Monochlorobiphenyls (% Recovery)	Tetrachlorobiphenyls (% Recovery)	Octachlorobiphenyls (% Recovery)	Decachlorobiphenyls (% Recovery)
Make Up Water				
1103	52	72	65	44[a]
1104	46[a]	75	70	44[a]
1105	62	80	75	50
1105 ms	34[a]	41[a]	36[a]	28[a]
1105 ms	67	87	79	60
1109	52	80	70	46[a]
1110 12:30	53	71	70	55
1110 14:45	54	71	69	55
1110 14:45	71	75	70	66
1110 16:30	62	57	55	44[a]
1111	65	60	59	46[a]
1112 11:50	32[a]	43[a]	47[a]	43[a]
1112 11:50 ms	58	85	89	96
1112 11:50 ms	52	77	83	95
1113 11:30	61	50	47[a]	38[a]
Mean	55	69	66	54
Standard Deviation	11	14	14	18

(Continued)

TABLE 8-5. (Continued)

Sample I. D.	Monochlorobiphenyls (% Recovery)	Tetrachlorobiphenyls (% Recovery)	Octachlorobiphenyls (% Recovery)	Decachlorobiphenyls (% Recovery)
Reagent blank	44	69	68	54
System blank	60	79	85	66
Reagent blank	64	78	83	71
Reagent blank	63	78	82	70
Reagent blank	66	80	83	56
Reagent blank	67	76	72	49[a]
Reagent blank	38[a]	66	70	53
System blank	80	66	62	66
Reagent blank	58	70	70	48[a]
Mean	60	74	75	59
Standard Deviation	12	5	8	9

[a] Outside accuracy objective (50-125% recovery) specified by the method.

The objectives for MS/MSD results were 50-125 percent recovery and 40 percent RPD. The results for MS/MSD for liquid samples are presented in Table 8-6. For samples 1103 and 1112 liquid, the recoveries for dichlorobiphenyl (46% and 49%) and pentachlorobiphenyl (47% and 43%) were slightly outside the accuracy objective, but were within the precision objective. Duplicate recoveries of all spiked compounds of Sample 1105 make-up water were outside the accuracy objective of RPD $\leq$40% specified by the method. This indicates poor repeatability for the sample matrix.

8.1.5 Duplicate Analysis of Liquid Samples

One duplicate analysis was performed for a liquid sample. Nothing was detected in either analysis, and the detection limits are at about the same level. The results are presented in Table 8-7.

8.1.6 Blank Sample Analyses of MM5-SASS and Ambient Air Samples

Blank sample results for EPA Method 5 source assessment sampling system (MM5-SASS) and ambient air samples are presented in Table 8-8. Two PCBs were detected in one reagent blank. They are listed below:

- Trichlorobiphenyl (96 ng); and
- Tetrachlorobiphenyl (450 ng).

There is evidence of contamination of field samples with PCBs resulting from the analytical process. Field samples with similar levels of tri- and tetrachlorobiphenyls should be viewed with care.

8.1.7 Spiked Sample Analyses for MM5-SASS and Ambient Air Samples

Surrogate spike recovery data for PCBs in MM5-SASS and ambient air samples are summarized in Table 8-9. Surrogate recoveries were outside the accuracy objective (50-125%) specified by the method for all four surrogate spike compounds for sample 1104 U and for all the ambient air PCP filters. Other results that are outside the accuracy objectives are presented below.

TABLE 8-6. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR LIQUID SAMPLES ANALYZED FOR PCBs

Compound	1103 Matrix Spike (%)	1103 Matrix Spike Duplicate (%)	RPD[a] (%)	1105 Matrix Spike (%)	1105 Matrix Spike Duplicate (%)	RPD[a] (%)	1112 11:50 Matrix Spike (%)	1112 11:50 Matrix Spike Duplicate (%)	RPD[a] (%)
Scrubber Effluent									
Monochlorobiphenyls	76	57	29[b]				65	74	13
Dichlorobiphenyls	54	46[c]	16				49[c]	49[c]	0
Trichlorobiphenyls	87	82	6				72	79	9
Tetrachlorobiphenyls	72	64	12				64	69	8
Pentachlorobiphenyls	51	47[c]	8				43[c]	52	19
Hexachlorobiphenyls	80	71	12				75	89	17
Heptachlorobiphenyls	106	97	9				105	125	17
Octachlorobiphenyls	93	84	10				98	105	7
Decachlorobiphenyls	75	72	4				86	97	12
Make Up Water									
Monochlorobiphenyls				44[c]	88	67[b]	61	61	0
Dichlorobiphenyls				33[c]	63	68[b]	51	46[c]	10
Trichlorobiphenyls				50	105	71[b]	88	77	13
Tetrachlorobiphenyls				43[c]	88	69[b]	80	68	16
Pentachlorobiphenyls				29[c]	63	74[b]	57	52	9
Hexachlorobiphenyls				44[c]	98	76[b]	101	90	12
Heptachlorobiphenyls				60	131[c]	74[b]	134	118	13
Octachlorobiphenyls				49[c]	109	76[b]	108	103	5
Decachlorobiphenyls				45[c]	93	70[b]	104	108	4

[a] Relative Percent Difference (RPD) (%) = $\frac{(\text{Maximum Value} - \text{Minimum Value})}{(\text{Maximum Value} + \text{Minimum Value})/2} \times 100.$

[b] Outside acceptance criteria (RPD $\leq$40%) specified by the method.

[c] Outside acceptance criteria (50 - 125% Recovery).

TABLE 8-7. REPLICATE ANALYSES OF LIQUID SAMPLES ANALYZED FOR PCBs

Compound	1110 1445 (ng/L)	1110 1445 (ng/L)	RPD (%)
Scrubber Water			
Monochlorobiphenyls	<30	<10	NC
Dichlorobiphenyls	<20	<30	NC
Trichlorobiphenyls	<50	<50	NC
Tetrachlorobiphenyls	<80	<80	NC
Pentachlorobiphenyls	<60	<60	NC
Hexachlorobiphenyls	<100	<100	NC
Heptachlorobiphenyls	<200	<200	NC
Octachlorobiphenyls	<200	<100	NC
Nonachlorobiphenyls	<200	<200	NC
Decachlorobiphenyls	<200	<200	NC

NC = Not Calculated.

TABLE 8-8. BLANK DATA REPORTED FOR MM5-SASS AND AMBIENT AIR SAMPLES ANALYZED FOR PCBs

Compound	PUF Blank (ng)	Reagent Blank (ng)	PUF Blank (ng)	Reagent Blank (ng)	Reagent Blank (ng)	PCS Blank (ng)	PCP Blank (ng)	Reagent Blank (ng)	System Blank (ng)
Monochlorobiphenyls	<40	<20	<20	<20	<30	<20	<20	<10	<10
Dichlorobiphenyls	<40	<20	<20	<20	<30	<20	<20	<10	<10
Trichlorobiphenyls	<80	<40	<40	96	<50	<40	<40	<20	<30
Tetrachlorobiphenyls	<100	<60	<60	450	<80	<70	<70	<30	<40
Pentachlorobiphenyls	<90	<40	<50	<40	<60	<50	<50	<20	<30
Hexachlorobiphenyls	<200	<80	<90	<90	<100	<90	<90	<40	<60
Heptachlorobiphenyls	<200	<100	<100	<100	<200	<100	<100	<70	<80
Octachlorobiphenyls	<200	<100	<100	<100	<200	<200	<200	<70	<90
Nonachlorobiphenyls	<300	<200	<200	<200	<200	<200	<200	<100	<100
Decachlorobiphenyls	<300	<200	<200	<100	<200	<200	<200	<100	<100

TABLE 8-9. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR MM5-SASS AND AMBIENT AIR SAMPLES ANALYZED FOR PCBs

Sample I.D.	Monochlorobiphenyls (% Recovery)	Tetrachlorobiphenyls (% Recovery)	Octachlorobiphenyls (% Recovery)	Decachlorobiphenyls (% Recovery)
PUF Filter				
1103 U	48[a]	63	62	53
1103 D	80	80	74	60
1104 U	24[a]	31[a]	28[a]	22[a]
1104 D	56	68	66	55
1105 U	56	66	69	64
1105 D	72	82	72	64
1106 U	NR	NR	NR	NR
1106 D	NR	NR	NR	NR
1109 U	52	61	61	47[a]
1109 D	45[a]	69	72	50
1110 U	73	77	69	62
1110 D	79	82	73	62
1111 U	77	85	78	62
1111 D	61	73	69	59
1112 U	63	73	79	89
1112 D	11[a]	79	81	73
1113 U	66	76	82	77
1113 D	57	40[a]	40[a]	33[a]
Mean	54	69	67	58
Standard Deviation	22	15	14	16

(Continued)

TABLE 8-9. (Continued)

Sample I.D.	Monochlorobiphenyls (% Recovery)	Tetrachlorobiphenyls (% Recovery)	Octachlorobiphenyls (% Recovery)	Decachlorobiphenyls (% Recovery)
Blank				
MS XAD Blank	100	80	75	85
MS XAD Blank	100	82	77	92
MS XAD Blank	84	75	71	78
MS XAD Blank	80	73	70	79
PUF Blank	51	70	74	56
PUF Blank	54	62	63	67
Reagent Blank	17[a]	68	64	68
Reagent Blank	81	62	61	66
Reagent Blank	NR	NR	NR	NR
Reagent Blank	66	65	67	73
Mean	62	68	67	70
Standard Deviation	24	5	5	8
Ambient Air				
1103 PCS	88	56	62	61
1103 PCP	43[a]	10[a]	13[a]	13[a]
1104 PCS	58	34[a]	44[a]	47[a]
1104 PCP	43[a]	37[a]	44[a]	54

(Continued)

TABLE 8-9. (Continued)

Sample I.D.	Monochlorobiphenyls (% Recovery)	Tetrachlorobiphenyls (% Recovery)	Octachlorobiphenyls (% Recovery)	Decachlorobiphenyls (% Recovery)
Ambient Air (Cont.)				
1105 PCS	100	74	76	78
1105 PCP	33[a]	22[a]	33[a]	40[a]
1109 PCS	NR	NR	NR	NR
1109 PCP	47[a]	37[a]	43[a]	44[a]
Reagent blank	81	70	68	70
System blank	82	72	71	67
Mean[b]	82	55	61	62
Standard Deviation[b]	27	20	16	16
Mean[c]	42	26	33	38
Standard Deviation[c]	6	13	14	18

[a] Outside accuracy objectives (50-125% recovery) specified by the method.

[b] Calculations for PCS filters.

[c] Calculations for PCP filters.

NR = Not Recovered. Sample diluted past surrogate recovery.

- Monochlorobiphenyl (48%, 45%, 11%, and 17%);
- Tetrachlorobiphenyl (40% and 34%);
- Octachlorobiphenyl (40% and 44%); and,
- Decachlorobiphenyl (47%, and 33%).

Matrix spike results for XAD blank samples analyzed for PCBs are summarized in Table 8-10. Matrix spike recoveries were outside the accuracy objectives (50-125%) specified in the method for the analytes presented below:

- Monochlorobiphenyl (40% and 39%);
- Dichlorobiphenyl (29% and 29%);
- Trichlorobiphenyl (44% and 45%);
- Tetrachlorobiphenyl (40% and 41%);
- Pentachlorobiphenyl (46%, and 28% and 29%);
- Hexachlorobiphenyl (49%); and,
- Decachlorobiphenyl (49%).

The recoveries for the second matrix spike XAD blank sample were generally half of what they should have been. An overall error like this may have been caused by adding a smaller spike amount that what was recorded.

Each of the matrix spike analyses was performed in duplicate. Precision estimates were based on the duplicate recovery data. Variability ranged from zero to 10 percent RPD. All recoveries were inside the QAPP accuracy objectives (RPD $\leq$20%). Recoveries for PCBs in MM5-SASS samples may be slightly low.

8.2 QA/QC Results for PCDD/PCDF Analyses

PCDDs and PCDFs were analyzed according to procedures outlined in EPA Method 8280. Holding time requirements were met. Mass calibration of the instrument was performed daily with PFTBA and verified using DFTPP ion abundance criteria and isotopic ratio criteria for PCDDs and PCDFs. Since selected-ion-monitoring mass spectrometry was used for PCDD/PCDF analysis,

TABLE 8-10. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE RECOVERIES FOR XAD BLANK SAMPLES ANALYZED FOR PCBs

Compound	XAD Blank Matrix Spike (%)	XAD Blank Matrix Spike Duplicate (%)	RPD[a] (%)	XAD Blank Matrix Spike (%)	XAD Blank Matrix Spike Duplicate (%)	RPD[a] (%)
Monochlorobiphenyls	75	80	6	40[b]	39[b]	3
Dichlorobiphenyls	51	56	9	29[b]	29[b]	0
Trichlorobiphenyls	76	81	6	44[b]	45[b]	2
Tetrachlorobiphenyls	68	74	8	40[b]	41[b]	2
Pentachlorobiphenyls	46[b]	50	8	28[b]	29[b]	4
Hexachlorobiphenyls	80	88	10	49[b]	51	4
Heptachlorobiphenyls	113	125	10	70	71	1
Octachlorobiphenyls	107	118	10	64	66	3
Decachlorobiphenyls	88	97	10	49[b]	50	2

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$

[b] Outside accuracy objective range (50-125% recovery) specified by the method.

retention time windows were determined for each isomer group by injection of a PCDD/PCDF retention time standard containing the first and last eluter from each isomer group. A TCDD chromatographic performance check mixture was analyzed daily to verify a minimum 25 percent valley resolution between 2,3,7,8-TCDD and 1,2,3,4-TCDD.

Response factors for each PCDD/PCDF isomer class were determined from a five point calibration, performed in triplicate. The average response factors for each isomer class were required to have a percent RSD less than 15 percent. Quantitation was based on a ^{13}carbon internal standard for each isomer class.

8.2.1 Blank Sample Analyses

No dioxins or furans were detected above the detection limit for solid or liquid samples. Blank sample results indicated no dioxin or furan contamination resulting from analysis.

8.2.2 Spiked Sample Analyses for Solid Samples

Surrogate spike recoveries for dioxin and furan analyses are summarized in Table 8-11 for solid samples. Isotope labeled dioxins and furans were used to check recovery and to provide an assessment of measurement variability. Recoveries for the dirt samples below were outside the accuracy objective (100±50%) specified by the lab:

- $^{13}C_{12}$-TCDD (43%, 48% and 152%);
- $^{13}C_{12}$-PeCDD (45%, 37%, 44%, 157%, 40% and 43%);
- $^{13}C_{12}$-HxCDD (34%, 21%, 42%, 43%, 97%, 47%, 48%, 27%, 30% and 40%);
- $^{13}C_{12}$-HpCDD (41%, 17%, 34%, 36%, 35%, 10%, 20% 0% and 40%);

TABLE 8-11. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR SOLID SAMPLES ANALYZED FOR PCDDs AND PCDFs

Sample I. D.	C13-TCDD (% Recovery)	C13-PeCDD (% Recovery)	C13-HxCDD (% Recovery)	C13-HpCDD (% Recovery)	C13-OCDD (% Recovery)	C13-TCDF (% Recovery)	C13PeCDF (% Recovery)	C13-HxCDF (% Recovery)	C13-HpCDF (% Recovery)	C13-OCDF (% Recovery)
Dirt										
1104	61	55	67	41[a]	59	62	62	39[a]	29[a]	17[a]
1104 (dup)	58	61	34[a]	ND	23[a]	61	61	35[a]	23[a]	18[a]
1105	91	51	54	71	15[a]	42[a]	49[a]	41[a]	33[a]	16[a]
1106	43[a]	51	21[a]	17[a]	13[a]	36[a]	31[a]	13[a]	9[a]	9[a]
1109 11:10	48[a]	45[a]	52	78	50	50	61	37[a]	28[a]	27[a]
1109 12:45 ad	83	37[a]	42[a]	56	29[a]	42[a]	46[a]	32[a]	22[a]	19[a]
1109 12:45 ad	92	53	43[a]	64	50	66	70	52	35[a]	37[a]
1109 17:00	114	88	79	97	116	114	133	80	117	92
1110 10:45	99	77	110	129	110	106	97	78	82	96
1110 12:45	120	89	88	34[a]	64	125	97	62	91	130
1110 14:50	51	ND	37[a]	ND	ND	60	61	55	ND	ND
1110 16:45	100	44[a]	47[a]	63	52	63	45[a]	35[a]	44[a]	41[a]
1111 10:30	67	93	54	36[a]	79	87	71	46[a]	32[a]	15[a]
1111 12:45	152[a]	157[a]	ND	ND	116	168[a]	166[a]	116	102	100
1111 14:50	77	63	48[a]	35[a]	17[a]	75	70	14[a]	17[a]	20[a]
1111 17:00	94	56	59	10[a]	12[a]	60	64	48[a]	29[a]	26[a]
1112 11:45	61	40[a]	27[a]	20[a]	14[a]	56	48[a]	6[a]	11[a]	15[a]
1112 13:45	70	56	62	0[a]	ND	56	42[a]	15[a]	0[a]	0[a]
1113 12:30 ad	73	43[a]	30[a]	ND	ND	52	43[a]	12[a]	ND	ND
1113 12:30 ad	100	59	40[a]	40[a]	62	102	66	13[a]	16[a]	40[a]
method blank	93	103	121	108	101	94	101	117	117	89
method blank	102	104	109	107	97	98	106	105	103	93
Ash										
1104	107	86	62	41[a]	26[a]	109	95	70	45[a]	31[a]
1105	94	73	55	37[a]	24[a]	97	84	67	43[a]	26[a]
1106	105	122	123	115	96	107	117	127	110	104
1109 11:00	85	88	91	83	61	89	91	92	83	66
1109 12:45 ad	72	79	82	86	66	78	82	63	58	65
1109 12:45 ad	100	98	100	91	81	103	105	68	60	82
1109 17:00	82	89	86	55	80	90	92	82	72	58
1110 10:45	83	94	95	81	98	85	93	78	66	95
1110 12:45	66	67	65	67	60	74	68	39[a]	66	48
1110 14:50	45[a]	56	52	48[a]	30[a]	44[a]	51	25[a]	38[a]	33[a]
1110 16:45	92	111	117	132	92	94	100	90	83	76

(Continued)

TABLE 8-11. (Continued)

Sample I. D.	C13-TCDD (% Recovery)	C13-PeCDD (% Recovery)	C13-HxCDD (% Recovery)	C13-HpCDD (% Recovery)	C13-OCDD (% Recovery)	C13-TCDF (% Recovery)	C13PeCDF (% Recovery)	C13-HxCDF (% Recovery)	C13-HpCDF (% Recovery)	C13-OCDF (% Recovery)
Ash (Cont.)										
1111 10:30	54	81	88	65	51	59	73	89	77	46
1111 12:45	68	87	68	44[a]	72	81	81	85	58	40
1111 14:50	0[a]	0[a]	0[a]	0[a]	0[a]	0[a]	0[a]	0[a]	0[a]	0[a]
1111 17:00	84	87	83	72	34[a]	85	88	87	69	52
1112 11:45	105	96	87	94	100	79	89	92	86	96
1112 13:45	91	97	124	172	90	91	102	110	107	97
1113 12:30 ad	73	81	82	83	65	74	79	82	73	74
1113 12:30 ad	76	98	118	118	119	80	97	112	123	114
method blank	56	67	86	73	73	60	67	78	86	56
method blank	89	99	109	97	85	91	100	105	100	78
method blank	103	78	60	47[a]	30[a]	101	87	60	42[a]	27[a]

[a] Outside accuracy objectives (100±50%) for surrogate recovery.

ND = Not Detected.

- $^{13}C_{12}$-OCDD (23%, 15%, 13%, 29%, 17%, 12% and 14%);
- $^{13}C_{12}$-TCDF (42%, 36%, 42% and 168%);
- $^{13}C_{12}$-PeCDF (49%, 31%, 46%, 45%, 166%, 48%, 42% and 43%);
- $^{13}C_{12}$-HxCDF (39%, 35%, 41%, 13%, 37%, 32%, 35%, 46%, 14%, 48%, 6%, 15%, 12% and 13%);
- $^{13}C_{12}$-HpCDF (29%, 23%, 33%, 9%, 28%, 22%, 35%, 44%, 32%, 17%, 29%, 11%, 0%, 0% and 16%); and
- $^{13}C_{12}$-OCDF (17%, 18%, 16%, 9%, 27%, 19%, 37%, 0%, 41%, 15%, 20%, 2%, 15%, 0%, 0% and 40%).

Recoveries for ash samples outside the acceptance criteria are listed below:

- $^{13}C_{12}$-TCDD (45%);
- $^{13}C_{12}$-HpCDD (41%, 37%, 48% and 44%);
- $^{13}C_{12}$-OCDD (26%, 24%, 30% and 34%);
- $^{13}C_{12}$-TCDF (44%);
- $^{13}C_{12}$-HxCDF (39% and 45%);
- $^{13}C_{12}$-HpCDF (39% and 25%); and
- $^{13}C_{12}$-OCDF (45%, 43%, 38% and 26%).

Recoveries for one method blank were outside the acceptance criteria for four surrogates:

- $^{13}C_{12}$-HpCDD (47%);
- $^{13}C_{12}$-OCDD (30%);
- $^{13}C_{12}$-HpCDF (42%); and
- $^{13}C_{12}$-OCDF (27%).

Spike recoveries for solid waste feed (dirt) were below the accuracy objectives (50-150% recovery) specified by the lab for approximately 25% of the samples. Results for dirt field samples may be biased low.

Spike recoveries were within the accuracy objective of 50-150 percent for most ash samples, with the exception of ash Sample 1111 12:45 (0% surrogate recovery). Surrogate spike recoveries of the higher weight dioxins and furans for the ash feed were occasionally below the 50-150 percent recovery spike recoveries. However, surrogate recoveries of blank samples for the dioxins and furans in this weight region ranged from 90-125 percent indicating acceptable analytical performance.

8.2.3 Duplicate Analysis of Solid Samples

Results for duplicate analysis of solid samples are presented in Table 8-12. Tetrachlorodibenzofuran was detected slightly above the detection limit in two pairs of samples. The relative percent difference for these pairs was zero. No other analytes were detected.

8.2.4 Spiked Sample Analysis of Liquid Samples

Results for spiked sample analysis of liquid samples are presented in Table 8-13. Recoveries for surrogate spikes of scrubber water outside the accuracy objectives (50-150%) specified by the lab are presented below:

- ${}^{13}C_{12}$-TCDD (35% and 44%);
- ${}^{13}C_{12}$-PeCDD (47%);
- ${}^{13}C_{12}$-HpCDD (48% and 39%);
- ${}^{13}C_{12}$-OCDD (49%, 48%, 48% and 45%);
- ${}^{13}C_{12}$-TCDF (33%, 45% and 36%);
- ${}^{13}C_{12}$-PeCDF (46%);
- ${}^{13}C_{12}$-HpCDF (39%); and
- ${}^{13}C_{12}$-OCDF (46%, 31%, 47% and 46%).

TABLE 8-12. REPLICATE ANALYSES OF SOLID SAMPLES ANALYZED FOR PCDDs AND PCDFs

Compound	1104 (ng/g)	1104 (ng/g)	RPD[a] (%)	1109 (ng/g)	1109 (ng/g)	RPD[a] (%)	1113 (ng/g)	1113 (ng/g)	RPD[a] (%)
Dirt									
Tetrachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Pentachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Hexachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Heptachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Octachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Tetrachlorodibenzofurans	0.06	0.06	0	0.04	0.08	0	0.05	ND	NC
Pentachlorodibenzofurans	ND	ND	NC	ND	ND	NC	ND	ND	NC
Hexachlorodibenzofurans	ND	ND	NC	ND	ND	NC	ND	ND	NC
Heptachlorodibenzofurans	ND	ND	NC	ND	ND	NC	ND	ND	NC
Octachlorodibenzofurans	ND	ND	NC	ND	ND	NC	ND	ND	NC
Ash									
Tetrachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Pentachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Hexachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Heptachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Octachlorodibenzo-p-dioxin	ND	ND	NC	ND	ND	NC	ND	ND	NC
Tetrachlorodibenzofurans	ND	ND	NC	ND	ND	NC	ND	ND	NC
Pentachlorodibenzofurans	ND	ND	NC	ND	ND	NC	ND	ND	NC
Hexachlorodibenzofurans	ND	ND	NC	ND	ND	NC	ND	ND	NC
Heptachlorodibenzofurans	ND	ND	NC	ND	ND	NC	ND	ND	NC
Octachlorodibenzofurans	ND	ND	NC	ND	ND	NC	ND	ND	NC

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}} \times 100.$

ND = Not Detected.
NC = Not Calculated.

TABLE 8-13. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR LIQUID SAMPLES ANALYZED FOR PCDDs AND PCDFs

Sample I. D.	C13-TCDD (% Recovery)	C13Pe-CDD (% Recovery)	C13-HxCDD (% Recovery)	C13-HpCDD (% Recovery)	C13-OCDD (% Recovery)	C13-TCDF (% Recovery)	C13PeCDF (% Recovery)	C13-HxCDF (% Recovery)	C13-HpCDF (% Recovery)	C13-OCDF (% Recovery)
Scrubber Effluent										
1104	100	134	170[a]	156[a]	117	103	127	159[a]	169[a]	151[a]
1105	88	78	68	63	56	89	83	72	62	55
1106	92	104	102	91	51	92	100	104	85	68
1109 10:45	78	73	74	57	49[a]	79	76	74	65	46[a]
1109 12:30	66	62	69	62	59	65	65	67	70	54
1109 17:00	25[a]	45[a]	65	49[a]	49[a]	25[a]	36	58	63	42[a]
1110 10:30	35[a]	54	71	61	62	33[a]	56	64	66	51
1110 14:45	44[a]	67	67	48[a]	58	45[a]	56	64	52	55
1111 10:15	102	107	104	114	92	97	104	105	104	103
1111 12:30	74	47[a]	51	39[a]	60	36[a]	46[a]	52	39[a]	31[a]
1111 14:45 ad	96	98	98	74	56	88	98	98	74	47[a]
1111 14:45 ad	100	101	94	88	69	98	109	103	83	57
1111 14:45	85	94	96	95	82	83	90	97	94	91
1111 16:30	97	101	98	86	83	97	111	99	79	65
1111 16:45	69	76	75	85	64	69	73	79	75	74
1112 11:45	84	98	110	102	82	88	94	108	104	97
1112 11:45 ad	68	97	112	91	78	68	86	99	90	71
1112 11:45 ad	66	73	97	71	48[a]	69	71	75	69	46[a]
1112 13:45	76	82	80	73	48[a]	75	83	89	73	54
1113 11:30 ad	62	88	101	96	88	64	77	99	102	90
1113 11:30 ad	75	107	116	114	100	77	101	93	95	94
1113 11:30 fdup	82	100	106	98	45[a]	88	103	112	95	61
method blank	60	85	85	70	59	57	74	78	73	53
method blank	108	118	73	48[a]	39[a]	102	98	72	53	40[a]
method blank	63	71	72	70	40[a]	91	70	76	64	47[a]
Make Up Water										
1104	98	74	59	47[a]	34[a]	100	84	68	51	35[a]
1105	91	86	87	69	64	89	91	57	52	54
1106	98	101	105	104	107	97	100	102	101	107
1109 10:45	65	63	66	60	57	63	64	66	67	41[a]
1110 14:45	52	67	69	54	46[a]	54	64	69	70	36[a]
1111 10:15	95	98	109	98	95	99	101	112	97	72
1112 11:50	66	73	97	71	48[a]	69	71	75	69	46[a]
1113 11:30	68	97	112	91	78	68	86	99	90	71

[a] Outside accuracy objective (100±50%) specified by the lab.

Recoveries for surrogate spikes of scrubber make up water outside the accuracy objectives are presented below:

- $^{13}C_{12}$-HpCDD (47%);
- $^{13}C_{12}$-OCDD (34%, 46% and 48%); and
- $^{13}C_{12}$-OCDF (35%, 41%, 36% and 46%).

Blank recoveries outside the accuracy objectives are presented below:

- $^{13}C_{12}$-HpCDD (48%);
- $^{13}C_{12}$-OCDD (39% and 40%); and
- $^{13}C_{12}$-OCDF (40% and 47%).

Average surrogate spike recoveries in scrubber water samples were within expected limits, with the exception of scrubber effluent samples 1104 and 1109 17:00. Recoveries were low for the make up water and blank higher molecular weight dioxins and furans.

8.2.5 Duplicate Analysis Results of Liquid Samples

No dioxins or furans were recovered above the detection limits, therefore no calculation could be made.

8.3 QA/QC Results for Volatile Organic Analyses

Samples for volatile organics were analyzed by scanning gas chromatography/mass spectrometry following EPA Method 8240. Analyte identification and quantitation were performed using response factors and retention times generated from a five point calibration curve, relative to the closest eluting of three internal standards. The mass spectrometer was tuned daily to give an acceptable spectrum for bromofluorobenzene (BFB). System performance was verified initially and after every 12 hours to ensure a minimum average response factor of 0.3 (0.25 for bromoform) for five system performance check compounds. A five point calibration, used for generating response factors, was performed initially using six calibration check compounds. A continuing calibration check was performed every 12 hours using the six calibration check compounds. The response factor for each calibration check compounds was required to agree within 25 percent of the average five-point response factor, or a new multipoint calibration curve was generated. Samples for Volatile Organic Analyses were extracted and analyzed within holding time requirements.

8.3.1 Blank Sample Analyses of Solid Samples

Blank sample analysis for solid samples is presented in Table 8-14. Methyl ethyl ketone, benzene, trichloroethene, 1,2-dibromo-3-chloropropane, trichloroethene, trichlorofluoromethane, and xylenes were detected in one or more blanks above the detection limit. Only trichloroethene (18 ug/g) was detected above the QAPP specified accuracy objective of 5 times the detection limit.

8.3.2 Spiked Sample Analyses of Solid Samples

Surrogate spike recovery data are summarized in Table 8-15 for volatile organic analyses of solid samples. Recovery for d4-1,2-dichloroethane (129%) was slightly above the accuracy objectives (70-120% recovery) specified by the method. However, these recoveries are not out of line with project requirements.

Matrix spike results for solid samples analyzed for volatile organics are summarized in Table 8-16. Matrix spike recoveries for all analytes in ash samples were within the accuracy objectives specified by Method 8240. Matrix spike analysis was not performed on a dirt sample. Repeatability of spike recovery data was within the laboratory objective of 50 percent RPD for all matrix spike duplicate results.

TABLE 8-14. BLANK DATA REPORTED ABOVE THE METHOD DETECTION LIMIT FOR SOLID SAMPLES ANALYZED FOR VOLATILES

Compound	Detection Limit (ug/g)	Type of Blank	Reported Value (ug/g)
Benzene	2.0	Extraction	2.4
Methyl Ethyl Ketone	25.0	Extraction	40
Methyl Ethyl Ketone	25.0	Extraction	54
Methyl Ethyl Ketone	25.0	Extraction	29
Trichloroethene	2.0	Extraction	18[a]
1,2-Dibromo-3-chloropropane	2.0	System	8.4
1,2-Dibromo-3-chloropropane	2.0	System	6.3
Trichloroethene	2.0	System	3.8
Trichloroethene	2.0	System	2.9
Trichlorofluoromethane	10.0	System	3.7
Xylenes	2.0	System	2.1
Xylenes	2.0	System	2.1

[a] Outside accuracy objective ($\leq$5 x DL) specified in the QAPP.

TABLE 8-15. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR SOLID SAMPLES ANALYZED FOR VOLATILES

Compound	d4-1,2-Dichloroethane (% Recovery)	d8-Toluene (% Recovery)	p-Bromofluorobenzene (% Recovery)
Dirt			
1103	121	95	104
1104	118	97	101
1105	126[a]	106	103
Mean	122	99	103
Standard Deviation	3	5	1
Ash			
1103	124[a]	101	107
1104	122[a]	89	118
1105	109	103	101
Mean	118	98	107
Standard Deviation	7	6	7
Reagent blank	116	96	97
Reagent blank	94	96	110
System blank	94	97	107
Mean	101	96	105
Standard Deviation	10	0	6

[a] Outside accuracy objective (70-121% recovery) specified in the method.

TABLE 8-16. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR SOLID SAMPLES ANALYZED FOR VOLATILES

Compound	1105 Matrix Spike (%)	1105 Matrix Spike Duplicate (%)	RPD[a] (%)
Dirt			
1,1-Dichloroethene	72	71	1
Toluene	107	97	10
Chlorobenzene	111	100	10
Benzene	90	86	5
Trichloroethene	81	84	4
Ash			
1,1-Dichloroethene	67	59	13
Toluene	92	91	1
Chlorobenzene	102	99	3
Benzene	83	87	5
Trichloroethene	75	103	31

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$

8.3.3 Blank Sample Analysis of Liquid Samples

No volatile compounds were detected above the detection limit for liquid samples. Blank sample results indicated no contamination from analysis.

8.3.4 Spiked Sample Analysis for Liquid Samples

Surrogate spike recovery data are summarized in Table 8-17 for volatile organic analyses of liquid samples. For sample 1104, recovery for d4-1,2-dichloroethane (129%) outside the accuracy objective (70-120%) specified in the method. This recovery is inside the mean plus 3 standard deviations. These results imply that field results may be biased slightly high for compounds impacted by this surrogate. Recovery of p-bromofluorobenzene (118%) for one system blank was outside the expected range of recovery (86-115%) specified in the method.

Recovery of matrix spike compounds was within the accuracy objectives specified in Method 8240 for the scrubber effluent sample that was spiked. Results are presented in Table 8-18. Matrix spike duplicate results were within the laboratory objective of 50 percent RPD for all matrix spike duplicate results.

8.3.5 Blank Sample Analysis of VOST Samples

No volatile compounds were detected above the detection limit for VOST samples. Blank sample results indicated no contamination from analysis.

8.3.6 Spiked Sample Analysis for VOST Samples

Surrogate spike recovery data are summarized in Table 8-19 for volatile organic analyses of VOST samples. For sample 1186, recovery for d4-1,2-dichloroethane (15%) was outside the accuracy objective (50-150%) specified

TABLE 8-17. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR LIQUID SAMPLES ANALYZED FOR VOLATILES

Compound	d4-1,2-Dichloroethane (% Recovery)	d8-Toluene (% Recovery)	p-Bromofluorobenzene (% Recovery)
Effluent			
1103	105	100	102
1104	121	97	100
1105	104	95	97
Mean	110	97	100
Standard Deviation	6	2	2
Make Up Water			
1103	106	101	94
1104	129[a]	99	104
1105	104	95	99
Mean	113	98	99
Standard Deviation	6	2	6
Blank			
System blank	82	110	118[b]
System blank	98	95	107
Mean	90	103	113
Standard Deviation	8	8	6

[a] Outside accuracy objective (70-121% recovery) specified in the method.

[b] Outside accuracy objective (86-115% recovery) specified in the method.

TABLE 8-18. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR LIQUID SAMPLES ANALYZED FOR VOLATILES

Compound	1105 Matrix Spike (%)	1105 Matrix Spike Duplicate (%)	RPD[a] (%)
Scrubber Effluent			
1,1-Dichloroethene	80	80	0
Toluene	115	116	1
Chlorobenzene	109	109	0
Benzene	98	92	6
Trichloroethene	79	83	5

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[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}}$ x 100.

TABLE 8-19. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR VOST ANALYSES

Compound	d8-Toluene (% Recovery)	d4-1,2-Dichloroethane (% Recovery)	p-Bromofluorobenzene (% Recovery)
1103	102	104	121
1103	105	111	120
1103	102	107	119
1104	102	97	115
1104	102	97	117
1105	102	99	114
1134	92	97	108
1135	84	94	130
1136	93	90	99
1137	97	96	122
1138	88	99	106
1141	101	91	93
1141	90	83	92
1163	101	91	93
1164	116	78	89
1173	93	96	102
1176	95	93	100
1183	119	63	71
1184	91	95	108
1185	94	57	83
1186	89	15[a]	81
1190	120	86	87
1193	113	79	68
Mean	100	88	102
Standard Deviation	10	20	17
Blank			
Laboratory	84	96	97
Laboratory	99	107	100
Laboratory	81	101	135
1132 (Field)	84	87	114
1177 (Field)	96	103	121
Mean	89	99	113
Standard Deviation	7	7	14

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[a] Outside accuracy objectives (50-150% recovery) specified in Method 5040.

in the method. Since this is one of four samples analyzed for 11/8, and the only one to be outside the accuracy objectives, this low recovery should not seriously affect the field sample recoveries.

8.4 QA/QC Results for Semivolatile Organics Analyses

Semivolatile extracts were analyzed using gas chromatography/mass spectrometry procedures described in EPA Method 8270. Identification and quantitation were performed using response factors and retention times generated from a five point calibration curve, relative to the closest eluting of six internal standards. The mass spectrometer was tuned daily to give an acceptable spectrum for DFTPP. System performance was verified initially and after every 12 hours to ensure a minimum average response factor of 0.050 using four system performance check compounds. A five point calibration, used for generating response factors, was performed initially using 13 calibration check compounds representing base/neutral and acid extractable organics. A continuing calibration check was performed every 12 hours using the calibration check compounds. The response factor for each calibration check compound was required to be within 30 percent of the average five-point response factor, or a new multipoint calibration curve was generated.

8.4.1 Blank Sample Analyses of Solid Samples

Blank sample results showed no detectable semivolatile organic contaminants resulting from sample collection, handling or analysis.

8.4.2 Spiked Sample Analyses for Solid Samples

Surrogate spike recovery data for semivolatile organics in solid samples are summarized in Table 8-20. These results show the mean surrogate recoveries within the accuracy objective specified in the method.

TABLE 8-20. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR SOLID SAMPLES ANALYZED FOR SEMIVOLATILES

COMPOUND	2-Fluorophenol (% Recovery)	Phenol-d5 (% Recovery)	Nitrobenzene-d5 (% Recovery)	2-Fluorobiphenol (% Recovery)	2,4,6-Tribromophenol (% Recovery)	Terphenyl-d14 (% Recovery)
Dirt						
1104	74	87	84	80	56	72
1105	77	85	74	88	52	52
1106	92	93	65	98	121	62
Mean	81	88	74	89	76	62
Standard Deviation	8	3	8	7	32	8
Ash						
1104	92	73	104	90	78	72
1105	77	32	88	91	52	65
1106	62	82	85	109	60	87
Mean	77	62	92	97	63	75
Standard Deviation	12	22	8	9	11	9
Blank						
Reagent Blank	69	81	83	107	96	88

8.4.3 Blank Sample Analyses of Liquid Samples

Blank sample results showed no detectable semivolatile organic contaminants resulting from sample collection, handling or analysis.

8.4.4 Spiked Sample Analyses for Liquid Samples

Surrogate spike recovery data for semivolatile organics in liquid samples is summarized in Table 8-21. These results show that results for most samples were within the accuracy objectives specified by the method. Recoveries of two surrogates (2-fluorophenol and phenol-d5) for scrubber effluent sample 1106 were outside the accuracy objectives specified in the method. Recovery for this field sample may be slightly low. Other recoveries outside the accuracy objectives were slightly high, but should not significantly affect the results.

Matrix spike results for liquid samples analyzed for semivolatile organics are summarized in Table 8-22. Matrix spike recoveries were within the accuracy objectives specified in the method for these analyses for all analytes except for pyrene. Recovery for pyrene was outside the accuracy objectives (52-115% recovery) for both scrubber effluent (118%) and make up water (119%) samples. These recoveries should not affect the field sample results.

Each of the matrix spike analyses was performed in duplicate. Precision estimates, based on the duplicate recovery data was very good (less than 20 percent RPD) for effluent samples. Variability was higher in make up water samples, ranging from one to 44 percent RPD. Recoveries were outside the QAPP accuracy objectives (RPD $\leq 20\%$) for three matrix spike compounds:

- 1,4-Dichlorobenzene (27%);
- Pentachlorophenol (44%); and
- p-Chloro-m-cresol (24%).

TABLE 8-21. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR LIQUID SAMPLES ANALYZED FOR SEMIVOLATILES

Compound	2-Fluorophenol (% Recovery)	Phenol-d5 (% Recovery)	Nitrobenzene-d5 (% Recovery)	2-Fluorobiphenol (% Recovery)	2,4,6-Tribromophenol (% Recovery)	Terphenyl-d14 (% Recovery)
Scrubber Effluent						
1104	62	63	75	63	37	59
1105	13[a]	6[b]	106	93	25	75
1106	39	12	69	69	31	53
Mean	38	27	83	75	31	62
Standard Deviation	20	26	16	13	5	9
Make up Water						
1104	82	98[b]	97	86	81	75
1105	106[a]	108[b]	103	107	103	89
Blank						
Reagent Blank	104[a]	104[b]	111	91	52	84
Reagent Blank	95	98[b]	98	99	80	100

[a] Outside accuracy objectives (10-94% recovery) specified by the method.

[b] Outside accuracy objectives (21-100% recovery) specified by the method.

TABLE 8-22. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR LIQUID SAMPLES ANALYZED FOR SEMIVOLATILES

COMPOUND Compound	1104	1104	RPD (%)
	--- (% Recovery) ---		
Scrubber Effluent			
Acenaphthene	85	91	7
p-Chloro-m-cresol	94	96	2
2-Chlorophenol	83	90	8
1,4-Dichlorobenzene	65	60	8
Di-n-propylnitrosamine	121	125	3
2,4-Dinitrotoluene	86	87	1
Nitrophenol	100	108	8
Pentachlorophenol	66	65	2
Phenol	86	93	8
Pyrene	99	118[b]	18
1,2,4-Tricholorobenzene	73	70	4
Make Up Water			
Acenaphthene	93	85	9
p-Chloro-m-cresol	110	87	24[c]
2-Chlorophenol	96	93	3
1,4-Dichlorobenzene	90	69	27[c]
Di-n-propylnitrosamine	117	110	6
2,4-Dinitrotoluene	103	104	1
Nitrophenol	94	116	21[c]
Pentachlorophenol	61	95	44[c]
Phenol	101	94	7
Pyrene	103	119[b]	14
1,2,4-Tricholorobenzene	85	82	4

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$

[b] Outside accuracy objectives (52-115% recovery) specified by the method.

[c] Outside accuracy objectives (RPD ≤20%) specified in the QAPP.

8.4.5 Blank Sample Analyses of MM5-SASS Samples

Blank sample results for MM5-SASS samples are presented in Table 8-23. Bis(2-ethylhexyl)phthalate and di-n-butylphthalate were detected in two samples. However, neither of these compounds were present at greater than five times the laboratory detection limit. Phthalates are common laboratory contaminants and the field sample results should not be greatly influenced by these results.

8.4.6 Spiked Sample Analyses for MM5-SASS Samples

Surrogate spike recovery data for semivolatile organics in MM5-SASS samples are summarized in Table 8-24. These results show that results for most samples were within the accuracy objectives specified by the method. Recoveries of three surrogates (2-fluorophenol, phenol-d5, 2,4,6-tribromophenol) for sample 1105 were outside the accuracy objectives specified in the method. Recovery for this field sample may be low. Other surrogate recoveries outside the accuracy objectives were slightly high, but should not significantly affect the results.

Matrix spike results for MM5-SASS samples analyzed for semivolatile organics are summarized in Table 8-25. Matrix spike recoveries were within the accuracy objectives specified in the method for these analyses for all analytes except for pentachlorophenol and phenol. Recovery for pentachlorophenol (9.6% and 1.7%) was outside the accuracy objectives (14-176% recovery) for the matrix spike and the duplicate. Recovery for phenol (120%) was outside the accuracy objectives (52-115% recovery) for only one of the matrix spike samples.

Each of the matrix spike analyses was performed in duplicate. Precision estimates were based on the duplicate recovery data. Variability ranged from two to 140 percent RPD. Recoveries were outside the QAPP accuracy objectives (RPD $\leq 20\%$) for six matrix spike compounds:

TABLE 8-23. BLANK DATA REPORTED ABOVE THE METHOD DETECTION LIMIT FOR MM5-SASS SAMPLES ANALYZED FOR SEMIVOLATILES

Compound	Detection Limit (ug)	1109 DXS Value (ug)	1109 DXP Value (ug)
Bis(2-ethylhexyl)phthalate	20.0	21.0	<20
Di-n-butylphthalate	20	69	57

TABLE 8-24. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR MM5-SASS SAMPLES ANALYZED FOR SEMIVOLATILES

Compound	2-Fluorophenol (% Recovery)	Phenol-d5 (% Recovery)	Nitrobenzene-d5 (% Recovery)	2-Fluorobiphenol (% Recovery)	2,4,6-Tribromophenol (% Recovery)	Terphenyl-d14 (% Recovery)
Filter						
1104 DXS	83	88	96	107	74	76
1105 DXP	3[a]	14[b]	41	87	10[c]	71
1105 DXS	73	91	86	99	57	6
1106 DXP	46	97	59	106	61	84
1106 DXS	93	98	85	105	105	90
Mean	60	78	73	101	61	78
Standard Deviation	32	32	20	7	31	8
Blank						
1109 DXS	128[a]	116[b]	94	103	37	85
1109 DXP	111	122[b]	95	120[d]	70	89
Reagent Blank	83	94	104	99	61	80

[a] Outside accuracy objectives (25-121% recovery) specified in the method.

[b] Outside accuracy objectives (24-113% recovery) specified in the method.

[c] Outside accuracy objectives (19-122% recovery) specified in the method.

[d] Outside accuracy objectives (43-116% recovery) specified in the method.

TABLE 8-25. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR XAD BLANK SAMPLES ANALYZED FOR SEMIVOLATILES

Compound	XAD Blank --- (% Recovery) ---		RPD[a] (%)
Filter			
Acenaphthene	103	75	32[b]
p-Chloro-m-cresol	111	85	27[b]
2-Chlorophenol	108	96	12
1,4-Dichlorobenzene	25	35	33[b]
Di-n-propylnitrosamine	88	90	2
2,4-Dinitrotoluene	71	86	19
Nitrophenol	31	71	78[b]
Pentachlorophenol	9.6[c]	1.7[c]	140[b]
Phenol	120[d]	101	17
Pyrene	113	101	11
1,2,4-Tricholorobenzene	81	62	27[b]

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$

[b] Outside accuracy objective (RPD $\leq$20%) specified in the QAPP.

[c] Outside accuracy objective (14-176% recovery) specified in the method.

[d] Outside accuracy objective (52-115% recovery) specified in the method.

- Acenaphthene (32%);
- p-Chloro-m-Cresol (27%);
- 1,4-Dichlorobenzene (33%);
- Nitrophenol (78%);
- Pentachlorophenol (140%); and
- 1,2,4-Trichlorobenzene (27%).

Poor repeatability for pentachlorophenol is probably due to the low recoveries for the matrix spike compounds.

8.5 QA/QC Results for Metals Analyses

Samples for metals were analyzed using inductively coupled plasma emission or atomic absorption spectroscopy, following procedures described in EPA Method 6010 (ICPES) or 7000 series (AAS). Calibration for ICPES analyses was based on a response factor generated daily for each metal using three exposures of a calibration standard and calibration blank. Calibration was verified by analyzing a QC check standard after every 10 samples, with agreement with ±10 percent of the expected value required for continuing analysis. Calibration for AAS analyses was based on a multipoint calibration curve generated daily for each metal using a calibration blank and three upscale standards. AAS calibration was verified after every ten samples by analyzing a QC check standard, with agreement within ±15 percent of the expected value required for continuing analysis.

8.5.1 Blank Sample Analyses for Solid Samples

Calibration blanks were analyzed every ten samples as part of the ongoing calibration verification procedure. Calibration blank results were required to be within five times the instrument detection limit, or the system was recalibrated. Laboratory reagent blanks were analyzed with each sample batch to assess background levels of analytes resulting from sample preparation, including addition of reagents and digestion. Reagent blank results for metals were subtracted from field sample results. Blank results are reported in Table 8-26. Beryllium, cadmium, chromium, copper, silver, vanadium and zinc were detected in the blank, but not above the acceptance criteria of 5 times the detection limit.

8.5.2 Spiked Sample Analyses for Solid Samples

Matrix spike/matrix spike duplicate results for metals analyses are summarized in Table 8-27 for solid samples. Samples of each matrix type were spiked with known concentrations of target metals. Each matrix spike analysis was performed in duplicate except for extraction blanks. Recoveries within 80-120 percent of the theoretical value were expected, with repeatability of the spike recovery within 20 percent RPD. Recoveries outside the acceptance window were flagged in the data reports to indicate inaccuracies due to matrix interferences. Method spikes, i.e., reagent blanks spiked and treated as a normal sample, were analyzed to verify analytical process control. Matrix spike results showed acceptable recoveries for all metals except:

- Selenium (1%, 0%, 38% and 0%);
- Barium (132% and 78%);
- Chromium (77%, 74% and 40%);
- Nickel (76%, 78% and 37%);
- Zinc (24%);
- Antimony (71%); and
- Cadmium (79%).

Selenium was not recovered in three of the four duplicate pairs of matrix spiked samples. Recovery of selenium in the fourth pair averaged 38%. Selenium results for field samples should be viewed with suspicion. Recovery of selenium in field samples may not reflect actual concentrations.

Overall, matrix spike duplicate results showed excellent repeatability, well within the precision objective of 20 percent RPD for most of the analyses. Results for matrix spike duplicate pairs were outside the limits for:

TABLE 8-26. BLANK DATA REPORTED ABOVE THE METHOD DETECTION LIMIT FOR SOLID SAMPLES ANALYZED FOR METALS

Element	Detection Limit (mg/L)	Analysis Date	Type	Result (mg/L)
Beryllium	0.001	1/29/88	Analytical	0.004
Cadmium	0.003	12/08/87	Analytical	0.009
Cadmium	0.003	1/29/88	Analytical	0.003
Cadmium	0.003	1/29/88	Analytical	0.005
Cadmium	0.003	1/29/88	Analytical	0.004
Cadmium	0.003	1/29/88	Analytical	0.006
Cadmium	0.003	1/29/88	Analytical	0.009
Chromium	0.009	1/29/88	Analytical	0.019
Chromium	0.009	1/29/88	Analytical	0.013
Chromium	0.009	1/29/88	Preparation	0.02
Copper	0.01	1/29/88	Analytical	0.014
Silver	0.009	1/29/88	Analytical	0.02
Silver	0.009	1/29/88	Analytical	0.009
Silver	0.009	1/29/88	Analytical	0.012
Silver	0.009	1/29/88	Analytical	0.012
Silver	0.009	1/29/88	Analytical	0.028
Vanadium	0.02	1/29/88	Analytical	0.022
Vanadium	0.02	1/29/88	Analytical	0.031
Vanadium	0.02	1/29/88	Analytical	0.044
Vanadium	0.02	1/29/88	Preparation	0.03
Zinc	0.006	1/29/88	Analytical	0.007
Zinc	0.006	1/29/88	Analytical	0.009
Zinc	0.006	1/29/88	Analytical	0.024
Zinc	0.006	1/29/88	Preparation	0.013
Zinc	0.006	1/29/88	Preparation	0.022

TABLE 8-27. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR SOLID SAMPLES ANALYZED FOR METALS

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (ug/g)	Spike Result (ug/g)	Spike Added (ug/g)	Recovery 1 (%)	Recovery 2 (%)	RPD^{a} (%)
Dirt								
12/08/87	1110 3	Barium	514	637	93.5	132^{b}	78^{b}	51^{c}
12/08/87	1110 3	Beryllium	<0.092	84.7	93.5	91	90	1
12/08/87	1110 3	Cadium	4	84.7	93.5	86	85	1
12/08/87	1110 3	Chromium	79	150	93.5	76^{b}	77^{b}	1
12/08/87	1110 3	Copper	15	95.2	93.5	86	87	1
12/08/87	1110 3	Nickel	32	103	93.5	76^{b}	76^{b}	0
12/08/87	1110 3	Silver	4.4	91.8	93.5	94	93	1
12/08/87	1110 3	Thallium	<8.3	78.8	93.5	84	88	5
12/08/87	1110 3	Vanadium	19	103	93.5	90	91	1
12/08/87	1110 3	Zinc	308	330	93.5	24^{b}	26^{b}	8
1/05/88	1110 2	Mercury	<0.05	1	1	100	105	5
1/05/88	1113	Mercury	0.12	1.05	1	93	93	0
1/25/88	1105	Antimony	1.3	4.8	5.1	79^{b}	63^{b}	23^{c}
1/11/88	1105	Selenium	<0.3	<0.3	1.9	2^{b}	0^{b}	200^{c}

(Continued)

TABLE 8-27. (Continued)

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (ug/g)	Spike Result (ug/g)	Spike Added (ug/g)	Recovery 1 (%)	Recovery 2 (%)	RPD^{a} (%)
Ash								
1/29/88	1111 3	Beryllium	<0.094	89.3	99	90	90	0
1/29/88	1111 3	Cadium	5.5	8939	99	85	86	1
1/29/88	1111 3	Chromium	76	146	99	71^{b}	76^{b}	7
1/29/88	1111 3	Copper	15	96.5	99	82	85	4
1/29/88	1111 3	Nickel	28	110	99	83	91	9
1/29/88	1111 3	Silver	3.5^{d}	100	99	97	97	0
1/29/88	1111 3	Thallium	<8.5	86.9	99	88	94	7
1/29/88	1111 3	Vanadium	24	107	99	84	87	4
1/29/88	1104	Beryllium	<0.097	83.5	93.5	89	89	0
1/29/88	1104	Cadium	5.5	85.1	93.5	85	90	6
1/29/88	1104	Chromium	76	146	93.5	72^{b}	76^{b}	5
1/29/88	1104	Copper	16	93.6	93.5	83	84	1
1/29/88	1104	Nickel	39	111	93.5	77^{b}	79^{b}	3
1/29/88	1104	Silver	3.1^{d}	92.5	93.5	96	98	2
1/29/88	1104	Thallium	<8.7	87.5	93.5	94	94	0
1/29/88	1104	Vanadium	34	112	93.5	83	86	4
1/05/88	1110 2	Mercury	<0.05	1	1	100	105	5
1/05/88	1113	Mercury	0.12	1.05	1	93	93	0
1/20/88	1103	Antimony	<0.3	4.1	4.9	84	116	32^{c}
1/20/88	1110 4	Antimony	<0.3	5.6	5	112	96	15
1/11/88	1103	Selenium	<0.3	<0.3	1.9	0^{b}	0^{b}	NC
1/11/88	1110 4	Selenium	<0.3	0.6	2	30^{b}	55^{b}	59^{c}

(Continued)

TABLE 8-27. (Continued)

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (ug/g)	Spike Result (ug/g)	Spike Added (ug/g)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Scrubber Solids								
12/08/87	1104	Beryllium	<0.1	89.5	96.2	93	93	0
12/08/87	1104	Cadium	37	113	96.2	79[b]	78[b]	1
12/08/87	1104	Chromium	228	272	96.2	46[b]	35[b]	27[c]
12/08/87	1104	Copper	108	191	96.2	86	80	7
12/08/87	1104	Nickel	242	291	96.2	51[b]	24[b]	72[c]
12/08/87	1104	Silver	6.5	82.6	96.2	82	89	8
12/08/87	1104	Thallium	<9	79.9	96.2	83	80	4
1/28/88	1104	Thallium	<8.3	93.8	90.9	103	96	7
12/08/87	1104	Vanadium	31	116	96.2	88	87	1
12/08/87	1103	Mercury	0.65	1.6	1	95	100	5
1/28/88	1104	Antimony	14	112	96	102	104	2
12/16/87	1104	Selenium	<0.28	<0.29	1.9	0[b]	0[b]	NC

(Continued)

TABLE 8-27. (Continued)

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (ug/g)	Spike Result (ug/g)	Spike Added (ug/g)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Blank								
12/08/87	Blank	Barium		0.88	1	88		
12/08/87	Blank	Beryllium		0.87	1	87		
12/08/87	Blank	Cadium		0.85	1	85		
12/08/87	Blank	Chromium		0.86	1	86		
12/08/87	Blank	Copper		0.88	1	88		
12/08/87	Blank	Nickel		0.84	1	84		
12/08/87	Blank	Silver		0.92	1	92		
12/08/87	Blank	Thallium		0.8	1	80		
1/28/88	Blank	Thallium		0.93	1	93		
12/08/87	Blank	Vanadium		0.83	1	83		
12/08/87	Blank	Zinc		0.85	1	85		
12/17/87	Blank	Lead		0.018	0.02	90		
1/28/88	Blank	Antimony		1.16	1	116		
12/16/87	Blank	Selenium		0.019	0.02	95		
1/29/88	Blank	Barium		0.88	1	88		
1/29/88	Blank	Beryllium		0.88	1	88		
1/29/88	Blank	Cadium		0.86	1	86		
1/29/88	Blank	Chromium		0.89	1	89		
1/29/88	Blank	Copper		0.85	1	85		
1/29/88	Blank	Nickel		0.88	1	88		

(Continued)

TABLE 8-27. (Continued)

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (ug/g)	Spike Result (ug/g)	Spike Added (ug/g)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Blank (Cont.)								
1/29/88	Blank	Silver		1.04	1	104		
1/29/88	Blank	Thallium		0.94	1	94		
1/29/88	Blank	Vanadium		0.87	1	87		
1/29/88	Blank	Zinc		0.87	1	87		
1/11/88	Blank	Arsenic		0.021	0.02	105		
1/11/88	Blank	Lead		0.023	0.02	115		
1/20/88	Blank	Antimony		0.059	0.05	116		
1/11/88	Blank	Selenium		0.023	0.02	115		

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[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}} \times 100.$

[b] Outside accuracy objective (100±20% Recovery) specified in the QAPP.

[c] Outside accuracy objective (RPD ≤20%) specified in the QAPP.

[d] Less than 5 times the detection limit.

NC= Not Calculated.

- Barium (51% RPD);
- Antimony (23% RPD and 32% RPD);
- Selenium (200% RPD and 59% RPD);
- Chromium (27% RPD); and
- Nickel (72% RPD).

8.5.3 Duplicate Analyses of Solid Samples

Duplicate analysis results are summarized in Table 8-28. Duplicate analyses for metals were performed on solid waste feed (dirt), ash, and scrubber solid samples. Precision, expressed as the RPD for duplicate analysis of samples split prior to digestion, ranged from zero to 88 percent for solid samples. The following elements did not meet the acceptance criteria for duplicate analysis:

- Zinc (21% and 47%);
- Barium (41%, 45%, and 26%);
- Chromium (28% and 30%);
- Lead (74% and 59%);
- Arsenic (88% and 26%);
- Antimony (34%);
- Nickel (50% and 48%); and
- Mercury (52%).

These precision estimates are typical for this matrix type, (e.g., larger variability for solid samples) and concentration levels.

8.5.4 Blank Analyses for Liquid Samples

Results for metals detected in the blank are presented in Table 8-29. Cadmium, chromium, vanadium, and zinc were the only metals detected in the blank above the detection limit. None of the recoveries exceeded the acceptance limits of 5 times the detection limit.

TABLE 8-28. REPLICATE ANALYSES OF SOLID SAMPLES ANALYZED FOR METALS

Analysis Date	Sample Name	Element	Result 1 (ug/g)	Result 2 (ug/g)	RPD[a] (%)
Dirt					
12/08/87	1110 2	Beryllium	<0.099	<0.085	NC
12/08/87	1110 2	Cadmium	3.9	4.4	12
12/08/87	1110 2	Silver	2[b]	1.6[b]	NC
12/08/87	1110 2	Nickel	20	21	5
12/08/87	1110 2	Zinc	246	305	21[c]
12/08/87	1110 2	Copper	16	16	0
12/08/87	1110 2	Thallium	<8.9	<7.7	NC
12/08/87	1110 2	Vanadium	23	22	4
12/08/87	1110 2	Barium	454	689	41[c]
12/08/87	1110 2	Chromium	65	88	30[c]
1/11/88	1110 4	Arsenic	11	11	0
1/11/88	1111 3	Selenium	<0.3	<0.3	0
1/11/88	1105	Selenium	<0.3	<0.3	NC
1/11/88	1105	Arsenic	12.5	12.7	NC
1/11/88	1105	Lead	590	608	1
1/11/88	1106	Lead	667	1450	3
1/11/88	1106	Selenium	<0.3	<0.3	74[c]
1/11/88	1106	Arsenic	9.8	9.8	NC
1/11/88	1106	Selenium	<0.3	<0.3	0
1/25/88	1110 2	Antimony	1.4[b]	1.4[b]	NC
1/25/88	1105	Antimony	5.1	4.4	NC
1/25/88	1106	Antimony	0.97[b]	1.2[b]	15
1/28/88	1103	Antimony	14	13	NC
1/05/88	1110 2	Mercury	<0.05	<0.05	7
1/05/88	1110 2	Mercury	1	1.05	NC
1/05/88	1113	Mercury	0.12	0.11	5
1/05/88	1113	Mercury	1.05	1.05	9
1/29/88	1110 3	Barium	637	581	0
1/29/88	1110 3	Beryllium	84.7	77	9
1/29/88	1110 3	Cadmium	84.7	76.5	9
1/29/88	1110 3	Chromium	150	145	10
1/29/88	1110 3	Copper	95.2	89.6	3
1/29/88	1110 3	Nickel	103	96.9	6
1/29/88	1110 3	Silver	91.8	84.1	6
1/29/88	1110 3	Thallium	78.8	75	8
1/29/88	1110 3	Vanadium	103	96.5	5
1/29/88	1110 3	Zinc	330	338	7
					2

(Continued)

TABLE 8-28. (Continued)

Analysis Date	Sample Name	Element	Result 1 (ug/g)	Result 2 (ug/g)	RPD[a] (%)
Ash					
1/11/88	1110 4	Lead	900	1020	12
1/11/88	1110 4	Arsenic	12.4	32	88[c]
1/11/88	1111 1	Arsenic	20	21	5
1/11/88	1111 1	Lead	1720	1680	2
1/11/88	1111 3	Lead	900	1020	12
1/11/88	1103	Lead	1030	1060	3
1/11/88	1103	Arsenic	18.3	20.8	13
1/11/88	1104	Arsenic	22	17	26[c]
1/11/88	1104	Lead	1690	920	59[c]
1/11/88	1105	Arsenic	14	14	0
1/11/88	1106	Lead	426	407	5
1/20/88	1109 3	Antimony	<0.3	<0.3	NC
1/20/88	1110 4	Antimony	5.6	4.8	15
1/20/88	1111 1	Antimony	<0.3	<0.3	NC
1/20/88	1103	Antimony	4.1	5.8	34[c]
1/20/88	1104	Antimony	<0.3	<0.3	NC
1/25/88	1112 2	Antimony	2	2.2	10
1/29/88	1111 3	Vanadium	26	26	0
1/29/88	1111 3	Chromium	82	74	10
1/29/88	1111 3	Thallium	<8.7	<8.1	NC
1/29/88	1111 3	Cadmium	7.7	7.5	3
1/29/88	1111 3	Silver	3.9[b]	3.6[b]	NC
1/29/88	1111 3	Beryllium	<0.097	<0.06	NC
1/29/88	1111 3	Copper	17	14	20
1/29/88	1111 3	Nickel	77	46	50[c]
1/29/88	1111 3	Zinc	388	335	15
1/29/88	1111 3	Barium	1160	894	26[c]
1/29/88	1103	Nickel	35	57	48[c]
1/29/88	1103	Copper	15	14	7
1/29/88	1103	Chromium	67	89	28[c]
1/29/88	1103	Vanadium	26	26	0
1/29/88	1103	Beryllium	<0.091	<0.093	NC
1/29/88	1103	Silver	3.2[b]	3[b]	NC
1/29/88	1103	Zinc	371	352	5
1/29/88	1103	Barium	1070	1690	45[c]
1/29/88	1103	Thallium	<8.2	<8.3	NC
1/29/88	1103	Cadmium	5.3	5.9	11
1/29/88	1104	Barium	947	1120	17
1/29/88	1104	Beryllium	83.5	78.5	6

(Continued)

TABLE 8-28. (Continued)

Analysis Date	Sample Name	Element	Result 1 (ug/g)	Result 2 (ug/g)	RPD[a] (%)
Ash (Cont.)					
1/29/88	1104	Cadmium	85.1	79.8	6
1/29/88	1104	Chromium	146	146	0
1/29/88	1104	Copper	93.6	90.4	4
1/29/88	1104	Nickel	111	109	2
1/29/88	1104	Silver	92.5	90.2	3
1/29/88	1104	Thallium	87.5	83.6	5
1/29/88	1104	Vanadium	112	110	3
1/29/88	1104	Zinc	697	432	47[c]
1/05/88	1110 2	Mercury	1.05	1	5
1/05/88	1113	Mercury	1	1.05	5
1/05/88	1113	Mercury	<0.05	<0.05	NC
1/11/88	1103	Selenium	<0.3	<0.3	NC
1/11/88	1104	Selenium	<0.3	<0.3	NC
1/11/88	1111 1	Selenium	0.6[b]	1.1[b]	NC
1/29/88	1111 3	Barium	870	810	7
1/29/88	1111 3	Beryllium	89.3	88.2	1
1/29/88	1111 3	Cadmium	89.9	89.6	0
1/29/88	1111 3	Chromium	146	150	3
1/29/88	1111 3	Copper	96.5	98.1	2
1/29/88	1111 3	Nickel	110	117	6
1/29/88	1111 3	Silver	100	98.8	1
1/29/88	1111 3	Thallium	86.9	92.3	6
1/29/88	1111 3	Vanadium	107	109	2
1/29/88	1111 3	Zinc	457	402	13
Scrubber Solids					
12/08/87	1103	Beryllium	<0.1	<0.09	NC
12/08/87	1103	Nickel	336	285	17
12/08/87	1103	Mercury	1.6	1.65	3
12/08/87	1103	Chromium	490	424	14
12/08/87	1103	Silver	8.5	7.1	18
12/08/87	1103	Copper	164	167	2
12/08/87	1103	Cadmium	31	31	0
12/08/87	1103	Thallium	<9	<8.5	NC
12/08/87	1103	Zinc	788	753	5
12/08/87	1103	Mercury	0.65	1.1	52[c]

(Continued)

TABLE 8-28. (Continued)

Analysis Date	Sample Name	Element	Result 1 (ug/g)	Result 2 (ug/g)	RPD[a] (%)
Scrubber Solids (Cont.)					
12/08/87	1103	Vanadium	34	34	0
12/08/87	1103	Barium	910	917	1
12/08/87	1104	Barium	1690	1600	5
12/08/87	1104	Beryllium	89.5	84.2	6
12/08/87	1104	Cadmium	113	108	5
12/08/87	1104	Chromium	272	260	5
12/08/87	1104	Copper	191	181	5
12/08/87	1104	Nickel	291	264	10
12/08/87	1104	Silver	82.6	87.4	6
12/08/87	1104	Thallium	79.9	72.4	10
12/08/87	1104	Vanadium	1160	1110	4
12/08/87	1104	Zinc	644	617	4
1/28/88	1103	Thallium	<9	<8.5	NC
1/28/88	1104	Antimony	112	109	3
12/16/87	1103	Selenium	<0.3	<0.29	NC
12/16/87	1104	Selenium	<0.28	<0.29	NC
12/16/87	1104	Selenium	<0.28	<0.28	NC
12/17/87	1103	Lead	1130	1210	7
12/17/87	1104	Lead	1050	1030	2
12/17/87	1104	Lead	860	906	5
12/18/87	1103	Arsenic	42	44	5
12/18/87	1104	Arsenic	35	37	6
1/28/87	1104	Thallium	93.8	92.8	1

[a] Relative Percent Difference (RPD) (%) =

$$\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$$

[b] Less than 5 times the method detection limit. Values in this range may vary as much 50-100% and therefore are not used for calculations.

[c] Outside accuracy objectives (RPD≤20%) specified in the QAPP.

NC = Not Calculated.
ND = Not Detected.

TABLE 8-29. BLANK DATA REPORTED ABOVE THE METHOD DETECTION LIMIT FOR LIQUID SAMPLES ANALYZED FOR METALS

Analysis Date	Element	Detection Limit (mg/L)	Type	Result (mg/L)
1/27/88	Cadmium	0.003	Analytical	0.003
1/27/88	Chromium	0.009	Analytical	0.01
1/27/88	Chromium	0.009	Analytical	0.019
1/27/88	Vanadium	0.02	Analytical	0.034
1/27/88	Vanadium	0.02	Analytical	0.042
1/27/88	Vanadium	0.02	Analytical	0.022
1/27/88	Zinc	0.006	Analytical	0.01
1/27/88	Zinc	0.006	Preparation	0.015

8.5.5 Spiked Sample Analysis for Liquid Samples

Matrix spike/matrix spike duplicate results for metals analyses are summarized in Table 8-30 for liquid samples analyses. Samples of each matrix type were spiked with known concentrations of target metals. Each matrix spike analysis was performed in duplicate except for extraction blanks. Recoveries within 80-120 percent of the theoretical value were expected, with repeatability of the spike recovery within 20 percent RPD. Recovery for selenium (48%) was low in the scrubber effluent sample. Recovery of matrix spike duplicate pairs was outside the 20 percent accuracy objective for arsenic (22% RPD).

8.5.6 Duplicate Analysis for Liquid Samples

Duplicate analysis results for liquid samples are presented in Table 8-31. Duplicate analysis was performed on scrubber effluent and makeup water. Only recovery of zinc in one sample was outside the 20 percent RPD accuracy objectives, showing good precision overall for these matrix types.

8.6 QA/QC Results for TCLP Extracts

TCLP Extracts were analyzed for volatiles, semivolatiles, and metals. The calibration requirements for these analyses are the same as those described in Sections 8.3, 8.4 and 8.5 of this report.

8.6.1 Blank Results for Volatiles in TCLP Extracts of Solids

Results for volatiles detected above the detection limit in blanks are presented in Table 8-32. None of the compounds detected in the blanks exceeded the accuracy objectives of 5 times the detection limit. Low level values in the field results for 1,1,2,2-tetrachloroethane, toluene, trichloroethene, and vinyl chloride may be from contamination and should be viewed with suspicion.

TABLE 8-30. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR LIQUID SAMPLES ANALYZED FOR METALS

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD^{a} (%)
Liquid								
1/27/88	1103	Barium	1.4	2.3	1	90	90	0
1/27/88	1103	Beryllium	<0.001	0.89	1	89	89	0
1/27/88	1103	Cadmium	0.044	0.91	1	87	86	1
1/27/88	1103	Chromium	0.47	1.32	1	85	88	3
1/27/88	1103	Copper	0.12	0.97	1	85	84	1
1/27/88	1103	Nickel	0.3	1.14	1	84	84	0
1/27/88	1103	Silver	0.01^{b}	0.95	1	94	93	1
1/27/88	1103	Thallium	<0.09	0.89	1	89	86	3
1/27/88	1103	Vanadium	0.086^{b}	1.01	1	92	89	3
1/27/88	1103	Zinc	0.56	1.4	1	84	83	1
1/18/88	1103	Arsenic	0.064	0.084	0.02	100	80	22^{c}
1/03/88	1103	Mercury	<0.0002	0.0036	0.004	90	90	0
1/03/88	1110 4	Mercury	0.0010^{b}	0.0048	0.004	95	100	5
1/26/88	1103	Antimony	0.14	0.19	0.05	100	100	0
1/18/88	1103	Selenium	0.019	0.029	0.02	50^{d}	45^{d}	11^{c}
Make Up Water								
1/27/88	1104	Barium	0.13	1.04	1	91	92	1
1/27/88	1104	Beryllium	<0.001	0.9	1	90	91	1
1/27/88	1104	Cadmium	<0.003	0.9	1	90	90	0
1/27/88	1104	Chromium	0.027^{b}	0.94	1	91	90	1

(Continued)

TABLE 8-30. (Continued)

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Make Up Water (Cont.)								
1/27/88	1104	Copper	<0.01	0.87	1	87	87	0
1/27/88	1104	Nickel	<0.02	0.89	1	89	90	1
1/27/88	1104	Silver	0.024[b]	0.94	1	92	96	4
1/27/88	1104	Thallium	<0.09	0.98	1	98	88	11
1/27/88	1104	Vanadium	0.045[b]	0.91	1	86	88	2
1/27/88	1104	Zinc	0.019[b]	0.9	1	88	88	0
1/19/88	1105	Arsenic	0.009[b]	0.027	0.02	90	100	11
1/18/88	1105	Lead	<0.001	0.022	0.02	110	120	9
1/3/88	1105	Mercury	<0.0002	0.0038	0.004	95	95	0
1/27/88	1105	Antimony	<0.003	0.056	0.05	112	112	0
1/18/88	1105	Selenium	<0.003	0.016	0.02	80	85	6
Blank								
1/27/88	Blank	Barium		0.89	1	89		
1/27/88	Blank	Beryllium		0.91	1	91		
1/27/88	Blank	Cadmium		0.92	1	92		
1/27/88	Blank	Chromium		0.92	1	92		
1/27/88	Blank	Copper		0.88	1	88		
1/27/88	Blank	Nickel		0.9	1	90		
1/27/88	Blank	Silver		1	1	100		

(Continued)

TABLE 8-30. (Continued)

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Blank (Cont.)								
1/27/88	Blank	Thallium		0.92	1	92		
1/27/88	Blank	Vanadium		0.92	1	92		
1/27/88	Blank	Zinc		0.9	1	90		
1/18/88	Blank	Arsenic		0.018	0.02	90		
1/18/88	Blank	Lead		0.022	0.02	110		
1/26/88	Blank	Antimony		0.059	0.05	118		
1/18/88	Blank	Selenium		0.02	0.02	100		

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}}$ x 100.

[b] Value less than 5 times the detection limit.

[c] Outside accuracy objective (RPD ≤20%) specified in the QAPP.

[d] Outside accuracy objective (100±20% recovery) specifed in the QAPP.

TABLE 8-31. REPLICATE ANALYSES OF LIQUID SAMPLES ANALYZED FOR METALS

Date	Sample Name	Element	Result 1 (mg/L)	Result 2 (mg/L)	RPD[a] (%)
Liquid					
1/03/88	1110 4	Mercury	0.001	0.0011	10
1/03/88	1110 4	Mercury	0.0048	0.005	4
1/03/88	1103	Mercury	0.0036	0.0036	0
1/03/88	1103	Mercury	<0.0002	<0.0002	NC
1/18/88	1111 1	Lead	0.5	0.48	4
1/18/88	1111 1	Selenium	0.003[b]	<0.003	NC
1/27/88	1103	Barium	2.3	2.3	0
1/27/88	1103	Beryllium	0.89	0.89	0
1/27/88	1103	Cadmium	0.91	0.9	1
1/27/88	1103	Chromium	1.32	1.35	2
1/27/88	1103	Copper	0.97	0.96	1
1/27/88	1103	Nickel	1.14	1.14	0
1/27/88	1103	Silver	0.95	0.94	1
1/27/88	1103	Thallium	0.89	0.86	3
1/27/88	1103	Vanadium	1.01	0.98	3
1/27/88	1103	Zinc	1.4	1.39	1
1/18/88	1103	Lead	1.5	1.4	7
1/18/88	1103	Arsenic	0.084	0.08	5
1/18/88	1103	Selenium	0.029	0.028	4
1/18/88	1104	Arsenic	0.06	0.061	2
1/18/88	1104	Lead	1.2	1.2	0
1/18/88	1104	Selenium	0.022	0.023	4
1/19/88	1110 1	Arsenic	0.026	0.026	0
1/19/88	1112 2	Arsenic	0.012[b]	0.013[b]	NC
1/26/88	1103	Antimony	0.19	0.19	0
1/26/88	1104	Antimony	0.2	0.2	0
1/26/88	1106	Antimony	0.38	0.37	3
1/27/88	1111 1	Antimony	0.13	0.13	0
1/27/88	1104	Nickel	0.25	0.24	4
1/27/88	1104	Silver	0.029	0.13[b]	NC
1/27/88	1104	Barium	1	1	0
1/27/88	1104	Cadium	0.077	0.082	6
1/27/88	1104	Zinc	0.46	0.45	2
1/27/88	1104	Thallium	<0.09	<0.09	NC
1/27/88	1104	Copper	0.083	0.077	8
1/27/88	1104	Chromium	0.34	0.33	3
1/27/88	1104	Vanadium	0.1	0.11	10
1/27/88	1104	Beryllium	<0.001	<0.001	NC

(Continued)

TABLE 8-31. (Continued)

Date	Sample Name	Element	Result 1 (mg/L)	Result 2 (mg/L)	RPD[a] (%)
Make Up Water					
1/03/88	1105	Mercury	0.0038	0.0038	0
1/18/88	1105	Lead	0.022	0.024	9
1/18/88	1105	Selenium	0.016	0.017	6
1/27/88	1105	Barium	1.04	1.05	1
1/27/88	1105	Beryllium	0.9	0.91	1
1/27/88	1105	Cadmium	0.9	0.9	0
1/27/88	1105	Chromium	0.94	0.93	1
1/27/88	1105	Copper	0.87	0.87	0
1/27/88	1105	Nickel	0.89	0.9	1
1/27/88	1105	Silver	0.94	0.98	4
1/27/88	1105	Thallium	0.98	0.88	11
1/27/88	1105	Vanadium	0.91	0.92	1
1/27/88	1105	Zinc	0.9	0.9	0
1/19/88	1105	Arsenic	0.027	0.029	7
1/19/88	1106	Arsenic	0.01[b]	0.01[b]	NC
1/19/88	1106	Arsenic	0.01[b]	0.01[b]	NC
1/27/88	1105	Antimony	0.056	0.056	0
1/27/88	1106	Copper	<0.01	<0.01	NC
1/27/88	1106	Lead	0.005	0.006	18
1/27/88	1106	Vanadium	0.024[b]	0.047[b]	NC
1/27/88	1106	Cadium	<0.003	0.003[b]	NC
1/27/88	1106	Barium	0.14	0.14	0
1/27/88	1106	Silver	0.021[b]	0.024[b]	NC
1/27/88	1106	Nickel	0.022[b]	<0.02	NC
1/27/88	1106	Antimony	<0.003	<0.003	NC
1/27/88	1106	Zinc	0.048	0.037	26[c]
1/27/88	1106	Chromium	<0.009	0.010[b]	NC
1/27/88	1106	Thallium	<0.09	<0.09	NC
1/27/88	1106	Beryllium	<0.001	<0.001	NC
1/27/88	1106	Selenium	<0.003	<0.003	NC

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$

[b] Value less than 5 times the detection limit. Recoveries in this range may vary by 50-100%, and therefore are not used for calculations.

[c] Outside accuracy objective (RPD $\leq$20%) specified in the QAPP.

NC = Not Calculated.

TABLE 8-32. BLANK DATA REPORTED ABOVE THE METHOD DETECTION LIMIT FOR TCLP EXTRACTS OF SOLID SAMPLES ANALYZED FOR VOLATILES

Compound	Detection Limit (ug/L)	Type of Blank	Blank Value (ug/L)
1,1,2,2-Tetrachloroethane	2.0	System	2.9
1,1,2,2-Tetrachloroethane	2.0	System	2.9
Toluene	2.0	TCLP	3.0
Toluene	2.0	TCLP	4.6
Trichloroethene	2.0	System	2.8
Trichloroethene	2.0	System	2.8
Trichloroethene	2.0	TCLP	8.4
Vinyl Chloride	2.0	TCLP	3.7

8.6.2 Spiked Sample Analysis for Volatiles in TCLP Extracts of Solids

Surrogate spike recovery data are summarized in Table 8-33 for volatile organic analyses of TCLP extracts of solid samples. These results show recoveries within the laboratory objective of 50-150 percent for all samples except one. Low recovery (45%) was reported for d4-1,2-dichloroethane in sample 1109 12:45.

8.6.3 Blank Results for Semivolatiles in TCLP Extracts of Solid

Blank sample results for TCLP extracts of solids showed no detectable semivolatile organic contaminants resulting from sample collection, handling or analysis.

8.6.4 Spiked Sample Analyses for Semivolatiles in TCLP Extracts of Solids

Surrogate spike recovery data for semivolatile organics in TCLP extracts of solid samples is summarized in Table 8-34. These results show the surrogate recoveries are within the accuracy objectives specified in the method except for recoveries for 2-fluorophenol (25-121% recovery). Recoveries outside the accuracy objective are presented below:

- 2-Fluorophenol (19%, 14%, 20%, 19%, 22%, 24%, 15%, 20%, 20%, 24%, 13%, 9%, 12%, and 24%);
- Phenol-d5 (6%, 20%, 14%, and 123%);
- Nitrobenzene-d5 (132%, 156%, and 172%); and
- 2,4,6-Tribromophenol (9%, and 21%).

Only recoveries of analytes impacted by 2-fluorophenyl may be affected by these recoveries.

TABLE 8-33. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR TCLP EXTRACTS OF SOLID SAMPLES ANALYZED FOR VOLATILES

Compound	d4-1,2-Dichloroethane (% Recovery)	d8-Toluene (% Recovery)	p-Bromofluorobenzene (% Recovery)
Ash			
1104	89	107	120
1105	85	112	139
1106	77	104	152
1109 1100	84	114	131
1109 1245	45	118	128
1109 1700	103	109	137
1110 1045	101	101	122
1110 1245	108	110	146
1110 1645	138	104	118
1111 1030	128	108	101
1111 1245	90	108	118
1111 1450	86	107	123
1112 1145	108	108	125
1112 1345	107	110	117
1113	93	115	114
Dirt			
1104	87	105	113
1105	80	108	130
1106	75	108	127
1109 1100	70	110	157[a]
1109 1245	81	106	143
1109 1700	82	121	131
1110 1045	85	102	133
1112 1145	119	106	134
1112 2	102	112	137
1113	91	103	133
Blank			
System Blank	82	110	118
System Blank	113	105	123
System Blank	113	97	126
System Blank	113	105	123
System Blank	82	110	118
TCLP Blank	96	107	114
TCLP Blank	104	107	134

[a] Outside accuracy objective (100 ± 50% recovery).

TABLE 8-34. SUMMARY OF SURROGATE SPIKED SAMPLE RECOVERIES FOR TCLP EXTRACTS

COMPOUND	2-Fluorophenol (% Recovery)	Phenol-d5 (% Recovery	Nitrobenzene-d5 (% Recovery)	2-Fluorobiphenol (% Recovery)	2,4,6-Tribromophenol (% Recovery)	Terphenyl-d14 (% Recovery)
Dirt						
1104	70	75	80	98	94	72
1105	54	58	61	77	76	58
1106	48	56	77	91	70	62
1109 1100	59	63	86	93	89	60
1109 1245	45	55	81	90	62	56
1109 1700	19[a]	30	69	80	9[b]	62
1110 1045	14[a]	24	71	76	24	60
1110 1245	20[a]	35	100	90	39	74
1110 1450	19[a]	46	132[c]	77	21[b]	73
1110 1645	22[a]	58	96	89	36	81
1111 1030	24[a]	62	102	91	36	88
1111 1245	27	72	106	101	29	105
1111 1450	26	58	96	97	54	80
1111 1700	25	44	102	105	38	98
1112 1	15[a]	24	66	85	76	85
1112 2	20[a]	28	71	91	85	90
1113	25	32	95	86	26	71
Mean	31	48	88	89	51	75
Standard Deviation	17	16	18	8	26	14

(Continued)

TABLE 8-34. (Continued)

COMPOUND	2-Fluorophenol (% Recovery)	Phenol-d5 (% Recovery	Nitrobenzene-d5 (% Recovery)	2-Fluorobiphenol (% Recovery)	2,4,6-Tribromophenol (% Recovery)	Terphenyl-d14 (% Recovery)
Ash						
1104	82	100	84	95	101	72
1105	51	6[d]	76	92	90	69
1106	60	58	79	90	85	68
1109 1100	29	50	85	97	70	71
1109 1245	37	61	98	93	72	76
1109 1700	28	48	95	94	39	82
1110 1045	20[a]	39	76	89	37	70
1110 1245	40	61	82	96	32	69
1110 1450	42	64	81	96	34	70
1110 1645	24[a]	40	87	102	61	86
1111 1030	69	72	114	95	49	74
1111 1245	47	67	101	105	62	90
1111 1450	41	42	76	86	84	72
1112 1	13[a]	20[d]	80	79	21	69
1112 2	9[a]	14[d]	156[c]	84	20	64
1113	12[a]	19	98	84	19	66
Mean	38	48	92	92	55	73
Standard Deviation	20	24	20	7	26	7

(Continued)

TABLE 8-34. (Continued)

COMPOUND	2-Fluorophenol (% Recovery)	Phenol-d5 (% Recovery	Nitrobenzene-d5 (% Recovery)	2-Fluorobiphenol (% Recovery)	2,4,6-Tribromophenol (% Recovery)	Terphenyl-d14 (% Recovery)
Blank						
Reagent Blank	95	97	99	95	58	97
TCLP Blank	56	62	172[c]	86	71	51
Reagent Blank	98	99	87	99	90	87
Reagent Blank	89	93	98	90	58	83
Reagent Blank	86	85	79	84	69	69
Method Blank	24[a]	41	96	101	38	86
Method Blank	95	104	97	88	96	81
TCLP Blank	111	108	105	69	45	74
Method Blank	110	123[d]	111	102	102	89
Mean	85	90	105	90	70	80
Standard Dev	26	23	25	10	21	13

[a] Outside accuracy objective (25-121% recovery) specified in the method.

[b] Outside accuracy objective (24-113% recovery) specified in the method.

[c] Outside accuracy objective (23-120% recovery) specified in the method.

[d] Outside accuracy objective (24-113% recovery) specified in the method.

Matrix spike results for TCLP extracts of solid samples analyzed for semivolatile organics are summarized in Table 8-35. Matrix spike recoveries were within the accuracy objectives specified in the method for these analyses for all most analytes. Recovery for 2,4-dinitrotoluene was outside the accuracy objectives (29-182% recovery) for one pair of samples, recovery for phenol (5-112%) was outside the accuracy objectives for one sample, and recovery for pyrene was outside the accuracy objectives (52-115% recovery) for one sample. Recoveries outside the accuracy objectives are listed below:

- 2,4-Dinitrotoluene (20% and 18%);
- Phenol (114%); and
- Pyrene (136%).

Each of the matrix spike analyses was performed in duplicate. Precision estimates, based on the duplicate recovery data was very good (less than 20 percent RPD) for most samples. Recoveries outside the QAPP accuracy objectives (RPD $\leq$20%) are listed below:

- 2-Chlorophenol (22%, 35%, and 22%);
- Di-n-propylnitrosamine (125%, 22%, and 36%);
- Nitrophenol (42%, and 23%);
- Pentachlorophenol (25%, and 99%);
- Phenol (24%, 22%, and 36%); and
- Pyrene (30%).

8.6.5 Blank Results for Metals in TCLP Extracts

Results for metals detected above the detection limit in blanks are presented in Table 8-36. Arsenic, barium, lead, cadmium, chromium and silver were detected in the matrix blank above the detection limit. Only one extraction blank for barium (0.15 mg/L) was outside the acceptance criteria of 0.045 mg/L.

TABLE 8-35. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR TCLP EXTRACTS OF SOLID SAMPLES ANALYZED FOR SEMIVOLATILES

COMPOUND	1105 (% Recovery)		RPD[a] (%)	1109 1700 (% Recovery)		RPD[a] (%)	1110 1045 (% Recovery)		RPD[a] (%)	1111 1245 (% Recovery)		RPD[a] (%)	1111 1450 (% Recovery)		RPD[a] (%)
Dirt															
Acenaphthene	83	97	16	65	69	6	84	87	4						
p-Chloro-m-cresol	98	113	14	57	60	5	74	67	10						
2-Chlorophenol	98	105	7	51	41	22[b]	40	57	35[b]						
1,4-Dichlorobenzene	73	75	3	81	79	3	96	95	1						
Di-n-propylnitrosamine	15	3.5	125[b]	58	59	2	64	80	22[b]						
2,4-Dinitrotoluene	80	78	3	68	73	7	100	89	12						
Nitrophenol	122	80	42[b]	20[c]	18[c]	11	42	35	18						
Pentachlorophenol	69	54	25[b]	38	38	0	55	49	12						
Phenol	107	114	6	47	37	24[b]	51	60	16						
Pyrene	90	91	1	92	91	1	115	113	2						
1,2,4-Tricholorobenzene	73	91	22[b]	92	97	5	116	111	4						
Ash															
Acenaphthene	110	85	26[b]							75	81	8	77	72	7
p-Chloro-m-cresol	120	91	28[b]							65	84	26[b]	80	68	16
2-Chlorophenol	94	80	16							62	77	22[b]	74	74	0
1,4-Dichlorobenzene	87	78	11							79	92	15	98	88	11
Di-n-propylnitrosamine	115	80	36[b]							65	70	7	78	70	11
2,4-Dinitrotoluene	101	114	12							84	89	6	88	83	6
Nitrophenol	72	57	23							85	87	2	88	70	23[b]
Pentachlorophenol	23	68	99[b]							59	56	5	57	49	15
Phenol	105	84	22[b]							51	73	36[b]	48	59	21[b]
Pyrene	136[e]	101	30[b]							109	108	1	107	98	9
1,2,4-Tricholorobenzene	98	97	1							107	102	5	108	94	14

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}}$ x 100.

[b] Outside accuracy objective (RPD $\leq$20%) specified in the QAPP.

[c] Outside accuracy objectives (29-182% recovery) specified in the method.

[d] Outside accuracy objectives (5-112% recovery) specified in the method.

[e] Outside accuracy objectives (52-115% recovery) specified in the method.

TABLE 8-36. BLANK DATA REPORTED ABOVE THE METHOD DETECTION LIMIT FOR TCLP EXTRACTS OF SOLID SAMPLES ANALYZED FOR METALS

Element	Detection Limit (mg/L)	Analysis Date	Type	Value (mg/L)
Arsenic	0.002	1/7/88	Extraction	0.006
Arsenic	0.002	1/7/88	Extraction	0.004
Barium	0.009	1/11/88	Digestion	0.009
Barium	0.009	1/22/88	Extraction	0.023
Barium	0.009	1/22/88	Extraction	0.15[a]
Barium	0.009	1/26/88	Extraction	0.021
Barium	0.009	1/26/88	Extraction	0.018
Barium	0.009	1/26/88	Extraction	0.02
Cadmium	0.003	1/26/88	Extraction	0.009
Cadmium	0.003	1/26/88	Extraction	0.003
Lead	0.002	1/22/88	Extraction	0.07
Lead	0.002	1/26/88	Digestion	0.07
Cadmium	0.003	1/26/88	Analytical	0.004
Cadmium	0.003	1/26/88	Analytical	0.004
Chromium	0.009	1/26/88	Analytical	0.017
Chromium	0.009	1/26/88	Analytical	0.012
Chromium	0.009	1/26/88	Analytical	0.012
Chromium	0.009	1/26/88	Analytical	0.012
Chromium	0.009	1/26/88	Analytical	0.012
Silver	0.009	1/26/88	Analytical	0.012
Silver	0.009	1/26/88	Analytical	0.012
Silver	0.009	1/26/88	Analytical	0.013

[a] Outside accuracy objective ($\leq$5 x MDL) specified in the QAPP.

8.6.6 Spiked Sample Results for Metals in TCLP Extracts of Solids

TCLP extracts were performed on ash and solid waste. Results for the matrix spikes of those extracts are presented in Table 8-37. Recovery outside the accuracy objectives of 100±20% in ash and dirt samples was reported for:

- Cadmium (79% and 79%);
- Barium (70%, 130% and 60%);
- Lead (0%, 72% and 71%);
- Selenium (0%, 0% and 0%); and
- Arsenic (150%).

High recovery was reported for silver in a dirt sample. Repeatability of the results for the following was outside the 20 percent accuracy objectives:

- Arsenic (40%);
- Barium (28%, 36% and 50%); and
- Lead.

For this matrix, these results are not unexpected.

8.6.7 Duplicate Sample Results for Metals in TCLP Extracts of Solids

Duplicate analysis results for metals in TCLP extracts of solids are presented in Table 8-38. Duplicate analysis was performed on solid waste and ash. Recoveries for lead (67%) and arsenic (40%) were outside the 20 percent RPD accuracy objectives, showing good precision for most elements for the TCLP matrix.

8.6.8 Spiked Sample Results for Metals in TCLP Extracts of Liquids

Results for matrix/matrix spike duplicates for metals in TCLP extracts of liquids are presented in Table 8-39. Again, results for selenium

TABLE 8-37. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR TCLP EXTRACTS OF SOLID SAMPLES ANALYZED FOR METALS

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Ash								
12/14/87	1111 2	Cadmium	0.004	0.79	1	79[b]	79[b]	0
12/14/87	1111 2	Chromium	0.14	0.96	1	82	82	0
12/14/87	1111 2	Barium	0.7	1.4	1	70[b]	70[b]	0
12/14/87	1111 2	Lead	0.053	0.75	1	70[b]	75[b]	7
12/14/87	1111 2	Mercury	<0.0002	0.0042	0.004	105	105	0
12/14/87	1111 2	Silver	0.01	0.88	1	87	88	1
12/14/87	1111 3	Cadmium	0.033	0.85	1	82	79[b]	4
12/14/87	1111 3	Barium	0.68	1.6	1	92	122[b]	28[c]
12/14/87	1111 3	Chromium	0.026	0.85	1	82	79[b]	4
12/14/87	1111 3	Mercury	<0.0002	0.004	0.004	105	109	4
12/14/87	1111 3	Silver	<0.009	0.92	1	92	90	2
12/14/87	1111 3	Lead	4.1	2.4	1	0[b]	0[b]	NC
12/16/87	1109 1	Arsenic	0.02	0.036	0.02	80	95	17
12/16/87	1109 1	Selenium	<0.003	<0.003	0.02	0[b]	0[b]	NC
12/31/87	1111 2	Arsenic	<0.004	0.03	0.02	150[b]	100	40[c]
12/31/87	1111 2	Arsenic	<0.004	0.016	0.02	80	90	12
1/04/88	1106	Selenium	<0.003	<0.003	0.02	0[b]	0[b]	NC
1/04/88	1106	Arsenic	0.04	0.02	0.036	0[b]	0[b]	NC
1/08/88	1104	Chromium	0.012	0.84	1	83	85	2
1/08/88	1104	Lead	<0.05	0.9	1	90	84	7
1/08/88	1104	Silver	0.016	0.84	1	82	83	1
1/08/88	1104	Cadmium	<0.003	0.81	1	81	82	1
1/08/88	1104	Barium	0.61	1.49	1	88	89	1

(Continued)

TABLE 8-37. (Continued)

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Dirt								
1/07/88	1109 3	Selenium	<0.015	<0.012	0.02	0[b]	0[b]	NC
1/07/88	1109 3	Arsenic	0.014	0.023	0.02	45[b]	50[b]	11
1/07/88	1110 1	Arsenic	<0.01	0.024	0.02	120	120	0
1/07/88	1110 1	Selenium	<0.015	<0.012	0.02	0[b]	0[b]	NC
1/26/88	1109 3	Silver	0.018	0.94	1	92	79[b]	15
1/26/88	1109 3	Barium	1.8	6.1	1	130[b]	90	36[c]
1/26/88	1109 3	Lead	0.89	1.6	1	71[b]	91	25[c]
1/26/88	1109 3	Cadmium	0.011	0.83	1	82	82	0
1/26/88	1109 3	Chromium	0.08	0.9	1	82	79[b]	4
1/26/88	1110 1	Barium	1.8	2.8	1	100	60[b]	50[c]
1/26/88	1110 1	Chromium	0.049	0.88	1	83	83	0
1/26/88	1110 1	Lead	0.66	1.7	1	104	84	21[c]
1/26/88	1110 1	Cadmium	0.012	0.84	1	83	82	1
1/26/88	1110 1	Silver	0.031	0.91	1	88	89	1

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}}$ x 100.

[b] Outside accuracy objective ($100 \pm 20\%$ recovery) specified in the QAPP.

[c] Outside accuracy objective ($\text{RPD} \leq 20\%$) specified in the QAPP.

ND = Not Detected.
NC = Not Calculated.

TABLE 8-38. REPLICATE ANALYSIS OF TCLP EXTRACTS OF SOLID SAMPLES ANALYZED FOR METALS

Sample Name	Type	Element	Result 1 (mg/L)	Result 1 (mg/L)	RPD[a] (%)
Ash					
1103	Preparation	Arsenic	<0.02	<0.02	NC
1103	Preparation	Mercury	<0.0002	<0.0002	NC
1103	Preparation	Selenium	0.003	<0.003	NC
1104	Matrix Spike	Barium	1.49	1.51	1
1104	Matrix Spike	Cadmium	0.81	0.82	1
1104	Matrix Spike	Chromium	0.84	0.86	2
1104	Matrix Spike	Lead	0.9	0.84	7
1104	Matrix Spike	Silver	0.84	0.85	1
1106	Matrix Spike	Arsenic	0.036	0.034	6
1106	Matrix Spike	Selenium	<0.003	<0.003	NC
1109 1	Matrix Spike	Arsenic	0.036	0.039	8
1109 1	Matrix Spike	Selenium	<0.003	<0.003	NC
1109 2	Matrix Spike	Arsenic	0.02	0.02	0
1109 2	Matrix Spike	Selenium	<0.003	<0.003	NC
1111 2	Matrix Spike	Barium	1.4	1.4	0
1111 2	Matrix Spike	Cadmium	0.79	0.79	0
1111 2	Matrix Spike	Chromium	0.96	0.94	2
1111 2	Matrix Spike	Lead	0.75	0.8	6
1111 2	Matrix Spike	Silver	0.88	0.89	1
1111 3	Matrix Spike	Barium	1.6	1.9	17
1111 3	Matrix Spike	Cadmium	0.85	0.82	4
1111 3	Matrix Spike	Chromium	0.85	0.82	4
1111 3	Matrix Spike	Lead	2.4	1.2	67[b]
1111 3	Matrix Spike	Silver	0.92	0.9	2
1111 2	Preparation	Mercury	<0.0002	<0.0002	NC
1111 2	Preparation	Mercury	<0.0002	<0.0002	NC
1111 2	Matrix Spike	Arsenic	0.03	0.02	40[b]
1111 2	Matrix Spike	Mercury	0.0042	0.0042	0
1111 2	Matrix Spike	Selenium	<0.003	<0.003	NC
1111 3	Matrix Spike	Arsenic	0.016	0.018	12
1111 3	Matrix Spike	Selenium	<0.003	<0.003	NC
1111 3	Matrix Spike	Mercury	0.0042	0.004	5

(Continued)

TABLE 8-38. (Continued)

Sample Name	Type	Element	Result 1 (mg/L)	Result 1 (mg/L)	RPD[a] (%)
Dirt					
1103	Preparation	Arsenic	<0.02	<0.02	NC
1103	Preparation	Selenium	<0.003	<0.003	NC
1109 3	Matrix Spike	Barium	3.1	2.7	14
1109 3	Matrix Spike	Cadmium	0.83	0.83	0
1109 3	Matrix Spike	Chromium	0.9	0.87	3
1109 3	Matrix Spike	Lead	1.6	1.8	12
1109 3	Matrix Spike	Silver	0.94	0.91	3
1110 1	Matrix Spike	Barium	2.8	2.4	15
1110 1	Matrix Spike	Cadmium	0.84	0.83	1
1110 1	Matrix Spike	Chromium	0.88	0.88	0
1110 1	Matrix Spike	Lead	1.7	1.5	13
1110 1	Matrix Spike	Silver	0.91	0.92	1
1110 2	Predigestion	Barium	2.2	2.2	0
1110 2	Predigestion	Cadmium	0.009[c]	0.007[c]	NC
1110 2	Predigestion	Chromium	0.087	0.072	19
1110 2	Predigestion	Lead	0.63	0.55	14
1110 2	Matrix Spike	Selenium	<0.012	<0.012	NC
1110 2	Predigestion	Silver	<0.009	0.018	NC
1111 1	Matrix Spike	Arsenic	<0.01	<0.01	NC
1111 1	Matrix Spike	Selenium	<0.012	<0.012	NC

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$

[b] Outside accuracy objective (RPD $\leq$20%) specified in the QAPP.

[c] Reported value less than 5 times the detection limit. Quantitation in this region can vary from 50-100%; the value is therefore not used for calculations.

NC = Not Calculated.

TABLE 8-39. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR TCLP EXTRACTS OF LIQUID SAMPLES ANALYZED FOR METALS

Analysis Date	Sample Name	Element	MATRIX SPIKES Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	MATRIX SPIKE DUPLICATES Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Liquid								
1/13/88	1104	Barium	0.23	1.18	1	95	95	0
1/13/88	1104	Cadmium	0.06	0.91	1	85	85	0
1/13/88	1104	Chromium	0.27	1.13	1	86	85	1
1/13/88	1104	Lead	<0.05	0.94	1	94	92	2
1/13/88	1104	Silver	<0.009	0.97	1	97	99	2
1/08/88	1112 2	Arsenic	0.009	0.03	0.02	105	105	0
1/11/88	1112 2	Barium	0.27	1.2	1	93	89	4
1/11/88	1112 2	Cadmium	<0.003	0.84	1	84	82	2
1/11/88	1112 2	Chromium	0.043	0.89	1	85	83	2
1/11/88	1112 2	Lead	<0.05	0.9	1	90	82	9
1/08/88	1112 2	Selenium	<0.003	0.013	0.02	65[b]	65[b]	0
1/11/88	1112 2	Silver	<0.009	0.94	1	94	88	7

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}} \times 100.$

[b] Outside accuracy objective (100$\pm$20% recovery) specified in the QAPP.

NC = Not Calculated.

(65%) are outside the accuracy objectives specified in the QAPP. Repeatability of these analysis was within the accuracy objective of 20 percent RPD for all elements.

8.6.9 Duplicate Sample Results for Metals in TCLP Extracts of Liquids

Duplicate analysis results for metals in TCLP extracts of liquids are presented in Table 8-40. Duplicate analysis was performed on scrubber effluent and blanks. All recoveries were well inside the 20 percent RPD accuracy objectives, showing excellent precision for these matrix types.

8.7 QA/QC Results for EP Extracts of Metals

EP Extracts were analyzed for metals. The calibration requirements for these analyses are the same as those described in Section 8.4 of this report.

8.7.1 Blank Results for EP Extracts of Metals

Results for metals detected above the detection limit in blanks are presented in Table 8-41. Cadmium and lead were detected in the blanks above the detection limit. Neither of these metals detected in the blanks was outside the accuracy objectives of 5 times the detection limit.

8.7.2 Spiked Sample Results for Metals in EP Extracts of Solids

Results for matrix/matrix spike duplicates for metals in EP extracts of solids are presented in Table 8-42. All the results are inside the accuracy objectives specified in the QAPP. Repeatability of these analysis was within the accuracy objective of 20 percent RPD for all elements.

TABLE 8-40. REPLICATE ANALYSIS OF TCLP EXTRACTS OF LIQUID SAMPLES ANALYZED FOR METALS

Sample Name	Type	Element	Result 1 (mg/L)	Result 1 (mg/L)	RPD[a] (%)
Liquid					
1104	Matrix Spike	Barium	1.18	1.18	0
1104	Matrix Spike	Cadmium	0.91	0.91	0
1104	Matrix Spike	Chromium	1.13	1.12	1
1104	Matrix Spike	Lead	0.94	0.92	2
1104	Matrix Spike	Silver	0.97	0.99	2
1105	Predigestion	Barium	0.69	0.71	3
1105	Predigestion	Cadmium	0.045	0.051	12
1105	Predigestion	Chromium	0.31	0.31	0
1105	Predigestion	Lead	<0.05	<0.05	NC
1105	Predigestion	Silver	<0.009	<0.009	NC
1112 2	Matrix Spike	Arsenic	0.03	0.03	0
1112 2	Matrix Spike	Barium	1.2	1.16	3
1112 2	Matrix Spike	Cadmium	0.84	0.82	2
1112 2	Matrix Spike	Chromium	0.89	0.87	2
1112 2	Matrix Spike	Lead	0.9	0.82	9
1112 2	Matrix Spike	Selenium	0.013	0.013	0
1112 2	Matrix Spike	Silver	0.94	0.88	7
Blank					
	Predigestion	Barium	0.018	0.018	0
	Predigestion	Cadmium	<0.003	<0.003	NC
	Predigestion	Chromium	<0.009	<0.009	NC
	Predigestion	Lead	<0.05	<0.05	NC
	Predigestion	Silver	<0.009	<0.009	NC

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}}$ x 100.

NC = Not Calculated.

TABLE 8-41. SUMMARY OF REAGENT BLANKS REPORTED ABOVE THE METHOD DETECTION LIMIT FOR EP EXTRACTS OF SAMPLES ANALYZED FOR METALS

Element	Detection Limit (mg/L)	Analysis Date	Type	Value (mg/L)
Cadmium	0.003	1/26/88	Extraction	0.003
Lead	0.002	1/22/88	Extraction	0.07

TABLE 8-42. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR EP EXTRACTS OF SOLID SAMPLES ANALYZED FOR METALS

Analysis Date	Sample Name	Element	MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
			Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Ash								
12/16/87	1109 1	Arsenic	<0.002	0.019	0.02	95	100	5
12/16/87	1109 1	Selenium	<0.003	<0.003	0.02	0	0	NC
12/30/87	1103	Mercury	<0.0002	0.0042	0.004	105	100	5
1/04/88	1103	Arsenic	<0.002	0.02	0.02	100	105	5
1/04/88	1103	Selenium	<0.003	<0.003	0.02	0	0	NC
1/08/88	1105	Silver	<0.009	0.98	1	98	96	2
1/08/88	1105	Silver	0.012	0.86	1	85	84	1
1/08/88	1105	Cadmium	0.009	0.86	1	85	84	1
1/08/88	1105	Lead	0.13	1.01	1	88	90	2
1/08/88	1105	Lead	0.25	1.18	1	93	87	7
1/08/88	1105	Chromium	0.092	0.91	1	82	83	1
1/08/88	1105	Barium	0.85	1.81	1	96	93	3
1/08/88	1105	Chromium	0.12	1	1	88	86	2
1/08/88	1105	Cadmium	0.008	0.83	1	82	82	0
1/08/88	1105	Barium	0.97	1.84	1	87	85	2
1/15/88	1112 1	Arsenic	0.002	0.02	0.02	90	95	5
1/15/88	1112 1	Selenium	<0.003	0.019	0.02	95	90	5
1/22/88	1112 1	Barium	0.012	1.57	1	91	91	0
1/22/88	1112 1	Silver	0.012	0.98	1	97	98	1
1/22/88	1112 1	Lead	0.022	1.87	1	99	93	6
1/22/88	1112 1	Chromium	0.037	0.87	1	85	97	13
1/22/88	1112 1	Cadmium	0.66	0.89	1	85	85	0

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}}$ x 100.

NC = Not Calculated.

8.7.3 Replicate Results for Metals in EP Extracts of Solids

Results for replicate analysis of metals in EP extracts of solids are presented in Table 8-43. All the results, except three pairs for chromium (26%, 21% and 34%) and one pair for cadmium (26%), are within the accuracy objectives (RPD ≤20%) specified in the QAPP. Four replicate analyses were performed for barium, cadmium, chromium, lead and silver. The coefficient of variation was within the accuracy objective (CV ≤20%) for all analyses.

8.7.4 Spiked Sample Results for Metals in EP Extracts of Liquids

Results for matrix/matrix spike duplicates for metals in EP extracts of liquids are presented in Table 8-44. All the results are inside the accuracy objectives (100±20% recovery) specified in the QAPP, except those for selenium which average 73%. Repeatability for all elements of these analyses was within the accuracy objective (RPD ≤20%) specified in the QAPP.

8.7.5 Duplicate Analysis Results of Metals in EP Extracts of Liquids

Results for replicate analysis of metals in EP extracts of solids are presented in Table 8-45. All the results are inside the accuracy objectives (RPD ≤ 20%) specified in the QAPP.

8.8 QA/QC Results for Metals in Method 5 and Ambient Air Samples

Samples from ambient air monitoring and Method 5 sampling were analyzed for metals. The calibration requirements for these analyses are the same as those described in Section 8.4 of this report.

TABLE 8-43. REPLICATE ANALYSIS OF EP EXTRACTS OF SOLID SAMPLES ANALYZED FOR METALS

Sample Name	Type of Replicate	Element	MATRIX SPIKES Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	MATRIX SPIKE DUPLICATES Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Ash								
1103	Preparation	Arsenic	0.02	0.021	5			
1103	Preparation	Mercury	0.0042	0.004	5			
1103	Preparation	Mercury	<0.0002	<0.0002	NC			
1103	Preparation	Selenium	<0.003	<0.003	NC			
1104	Preparation	Arsenic	<0.002	<0.002	NC			
1104	Preparation	Barium	0.67	0.68	1			
1104	Preparation	Cadmium	0.003[c]	0.009[c]	NC			
1104	Preparation	Chromium	0.015	0.014	7			
1104	Preparation	Lead	<0.05	<0.05	NC			
1104	Preparation	Selenium	<0.003	<0.003	NC			
1104	Preparation	Silver	<0.009	<0.009	NC			
1105	Preparation	Arsenic	<0.002	<0.002	NC			
1105	Preparation	Barium	0.97	0.85	13			
1105	Preparation	Cadmium	0.008[c]	0.009[c]	NC			
1105	Preparation	Chromium	0.092	0.12	26[d]			
1105	Preparation	Lead	0.13[c]	0.25[c]	NC			
1105	Preparation	Mercury	<0.0002	<0.0002	NC			
1105	Preparation	Selenium	<0.003	<0.003	NC			
1105	Preparation	Silver	0.012	<0.009	NC			
1105	Preparation	Barium	1.84	1.82	1	1.81	1.78	1
1105	Preparation	Cadmium	0.83	0.83	0	0.86	0.85	1
1105	Preparation	Chromium	0.91	0.92	1	1	0.98	4
1105	Preparation	Lead	1.01	1.03	2	1.18	1.12	6
1105	Preparation	Silver	0.86	0.85	1	0.98	0.96	6
1109 1	Preparation	Arsenic	0.019	0.02	5			
1109 1	Preparation	Selenium	0.006[c]	0.009[c]	NC			

(Continued)

TABLE 8-43. (Continued)

Sample Name	Type of Replicate	Element	MATRIX SPIKES Sample Result (mg/L)	MATRIX SPIKES Spike Result (mg/L)	MATRIX SPIKES Spike Added (mg/L)	MATRIX SPIKE DUPLICATES Recovery 1 (%)	MATRIX SPIKE DUPLICATES Recovery 2 (%)	RPD[a] (%)
Ash (Cont.)								
1109 1	Preparation	Barium	0.7	0.7	0			
1109 1	Preparation	Cadmium	0.015[c]	0.021	NC			
1109 1	Preparation	Chromium	<0.009	<0.009	NC			
1109 1	Preparation	Lead	4.1	4	2			
1109 1	Preparation	Silver	0.017	<0.009	NC			
1109 2	Preparation	Arsenic	<0.002	0.002[c]	NC			
1109 2	Preparation	Selenium	<0.003	<0.003	NC			
1110 1	Preparation	Barium	0.98	0.98	0			
1110 1	Preparation	Barium	1.1	1.1	0			
1110 1	Preparation	Cadmium	<0.003	0.005[c]	NC			
1110 1	Preparation	Cadmium	0.012	0.011	9			
1110 1	Preparation	Chromium	0.099	0.097	2			
1110 1	Preparation	Chromium	0.025	0.025	0			
1110 1	Preparation	Lead	0.41	0.45	9			
1110 1	Preparation	Lead	0.15	0.14	7			
1110 1	Preparation	Silver	0.03	0.018[c]	NC			
1110 1	Preparation	Silver	0.017	0.018	6			
1112 1	Preparation	Arsenic	0.02	0.021	5			
1112 1	Preparation	Barium	1.57	1.57	0			
1112 1	Preparation	Cadmium	0.89	0.89	0			
1112 1	Preparation	Chromium	0.87	0.89	2			
1112 1	Preparation	Lead	1.87	1.81	3			
1112 1	Preparation	Selenium	0.019	0.018	5			
1112 1	Preparation	Silver	0.98	0.99	1			
1112 2	Preparation	Arsenic	0.004	0.004	0			

(Continued)

TABLE 8-43. (Continued)

Sample Name	Type of Replicate	Element	MATRIX SPIKES Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	MATRIX SPIKE DUPLICATES Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Ash (Cont.)								
1112 2	Preparation	Barium	0.81	0.8	1			
1112 2	Preparation	Cadmium	0.026	0.02[c]	26[d]			
1112 2	Preparation	Chromium	0.021[c]	0.009[c]	NC			
1112 2	Preparation	Lead	0.9	0.89	1			
1112 2	Preparation	Selenium	<0.003	<0.003	NC			
1112 2	Preparation	Silver	0.009	<0.009	NC			
Dirt								
1103	Preparation	Barium	0.96	0.97	1			
1103	Preparation	Barium	0.83	0.96	15			
1103	Preparation	Cadmium	<0.003	<0.003	NC			
1103	Preparation	Cadmium	0.009[c]	<0.003	NC			
1103	Preparation	Chromium	0.069	0.056	21[d]			
1103	Preparation	Chromium	0.049	0.069	34[d]			
1103	Preparation	Lead	0.25	0.27	8			
1103	Preparation	Lead	0.14[c]	0.25[c]	NC			
1103	Preparation	Silver	<0.009	<0.009	NC			
1103	Preparation	Silver	0.02	<0.009	NC			
1109 1	Preparation	Arsenic	<0.002	<0.002	NC			
1109 1	Preparation	Barium	0.93	0.94	1			
1109 1	Preparation	Cadmium	0.003[c]	0.006[c]	NC			
1109 1	Preparation	Chromium	0.047	0.052	10			

(Continued)

TABLE 8-43. (Continued)

			MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
Sample Name	Type of Replicate	Element	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
Dirt (Cont.)								
1109 1	Preparation	Lead	<0.05	0.052	NC			
1109 1	Preparation	Selenium	<0.003	<0.003	NC			
1109 1	Preparation	Silver	0.01	0.01	0			
1111 2	Preparation	Arsenic	<0.002	<0.002	NC			
1111 2	Preparation	Selenium	<0.003	<0.003	NC			
1112 1	Preparation	Mercury	<0.0002	<0.0002	NC			
1112 1	Preparation	Mercury	0.0038	0.0042	10			

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}}$ x 100.

[b] Coefficient of Variation (CV) (%) = Standard Deviation/Mean x 100.

[c] Reported value is less than 5 times the detection limit. Quantitation in this region can vary from 50-100%. These values are not used for calculations.

[d] Outside accuracy objective (RPD $\leq$ 20%) specified in the QAPP).

ND = Not Calculated.

TABLE 8-44. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR EP EXTRACTS OF LIQUID SAMPLES ANALYZED FOR METALS

			MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
Analysis Date	Sample Name	Element	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
12/31/87	1111 1	Mercury	<0.0002	0.0038	0.0004	95	95	0
1/02/88	1103	Mercury	<0.0002	0.0038	0.004	95	95	0
1/06/88	1103	Arsenic	0.032	0.052	0.02	95	100	5
1/06/88	1103	Selenium	0.012	0.032	0.02	100	100	0
1/08/88	1111 4	Arsenic	0.015	0.036	0.02	100	105	5
1/08/88	1111 4	Selenium	<0.012	0.02	0.015	75[b]	70[b]	7
1/11/88	1111 4	Silver	<0.009	0.93	1	93	92	1
1/11/88	1111 4	Barium	0.3	1.23	1	93	89	4
1/11/88	1111 4	Cadmium	0.009	0.87	1	86	83	4
1/11/88	1111 4	Lead	<0.05	0.91	1	91	88	3
1/11/88	1111 4	Chromium	0.094	0.96	1	87	83	5
1/08/88	Blank	Silver		0.99	1	99		
1/08/88	Blank	Barium		0.92	1	92		
12/16/87	Blank	Arsenic		0.017	0.02	85		
1/06/88	Blank	Arsenic		0.018	0.02	90		
12/14/87	Blank	Barium		0.88	1	88		
1/07/88	Blank	Selenium		0.021	0.02	110		
12/14/87	Blank	Silver		0.92	1	92		
1/04/88	Blank	Selenium		0.022	0.02	110		
12/14/87	Blank	Chromium		0.85	1	85		
1/08/88	Blank	Cadmium		0.88	1	88		
12/14/87	Blank	Cadmium		0.88	1	88		
1/08/88	Blank	Lead		0.9	1	90		
1/11/88	Blank	Cadmium		0.88	1	88		

(Continued)

TABLE 8-44. (Continued)

			MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
Analysis Date	Sample Name	Element	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
12/14/87	Blank	Lead		0.84	1	84		
1/11/88	Blank	Lead		0.94	1	94		
1/08/88	Blank	Silver		0.88	1	88		
1/11/88	Blank	Barium		0.88	1	88		
1/08/88	Blank	Lead		0.98	1	98		
1/11/88	Blank	Silver		0.96	1	96		
1/04/88	Blank	Arsenic		0.017	0.02	85		
1/11/88	Blank	Chromium		0.9	1	90		
1/07/88	Blank	Arsenic		0.018	0.02	90		
1/15/88	Blank	Selenium		0.021	0.02	105		
1/08/88	Blank	Barium		0.86	1	86		
1/15/88	Blank	Arsenic		0.019	0.02	95		
1/08/88	Blank	Cadmium		0.9	1	90		
1/22/88	Blank	Chromium		0.87	1	87		
1/08/88	Blank	Chromium		0.9	1	90		
1/22/88	Blank	Lead		0.95	1	95		
12/31/87	Blank	Arsenic		0.017	0.02	85		
1/22/88	Blank	Silver		0.99	1	99		
1/08/88	Blank	Arsenic		0.021	0.02	105		
1/08/88	Blank	Chromium		0.85	1	85		
1/06/88	Blank	Selenium		0.024	0.02	120		
1/22/88	Blank	Cadmium		0.9	1	90		
1/22/88	Blank	Barium		0.89	1	89		

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[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}}$ x 100.

[b] Outside accuracy objective (100±20% recovery) specified in the QAPP.

TABLE 8-45. REPLICATE ANALYSIS OF EP EXTRACTS OF LIQUID SAMPLES ANALYZED FOR METALS

Sample Name	Type	Element	Result 1 (mg/L)	Result 2 (mg/L)	RPD[a] (%)
1103	Preparation	Mercury	<0.0002	<0.0002	NC
1103	Preparation	Mercury	0.0038	0.0038	0
1104	Matrix Spike	Arsenic	0.051	0.052	2
1104	Preparation	Arsenic	0.022	0.022	0
1104	Preparation	Selenium	0.024	0.024	0
1104	Matrix Spike	Selenium	0.032	0.032	0
1111 1	Preparation	Mercury	<0.0002	<0.0002	NC
1111 3	Matrix Spike	Arsenic	0.015	0.016	6
1111 3	Preparation	Barium	0.29	0.29	0
1111 3	Preparation	Cadmium	0.013[b]	0.011[b]	NC
1111 3	Preparation	Chromium	0.12	0.11	9
1111 3	Preparation	Lead	0.07[b]	0.056[b]	NC
1111 3	Matrix Spike	Selenium	<0.003	<0.003	NC
1111 3	Preparation	Silver	<0.009	<0.009	NC
1111 4	Matrix Spike	Selenium	0.015	0.014	7
1111 4	Matrix Spike	Arsenic	0.035	0.036	3
1111 4	Matrix Spike	Barium	1.23	1.19	3
1111 4	Matrix Spike	Cadmium	0.87	0.84	4
1111 4	Matrix Spike	Chromium	0.96	0.92	4
1111 4	Matrix Spike	Lead	0.91	0.88	3
1111 4	Matrix Spike	Silver	0.93	0.92	1
1111 1	Matrix Spike	Mercury	0.0038	0.0038	0
1113	Matrix Spike	Arsenic	0.01	0.01	0
1113	Preparation	Barium	0.067	0.064	5
1113	Preparation	Cadmium	<0.003	0.008	NC
1113	Preparation	Chromium	0.053	0.055	4
1113	Preparation	Lead	<0.05	<0.05	NC
1113	Matrix Spike	Selenium	0.009	0.009	0
1113	Preparation	Silver	<0.009	<0.009	NC
Blank					
	Analytical	Selenium	<0.003	<0.003	NC

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[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$

[b] Reported values are less than 5 times the method detection limit. Values in this range may vary as much as 50-100% and are not used for these calculations.

NC = Not Calculated.

8.8.1 Blank Results for Metals in Method 5 and Ambient Air Samples

Results for metals detected in blank samples are presented in Table 8-46. Cadmium, chromium, nickel, silver, vanadium, and zinc were detected in trace quantities in the laboratory blanks. These results were still within the accuracy objectives (5 times the detection limit) specified in the QAPP.

Lead, mercury, antimony, and selenium were detected in the field blank for the Method 5 train in greater than trace quantities. Field samples with concentrations of these elements at those levels may be viewed with suspicion. A field blank was not taken for the ambient air filters.

8.8.2 Spiked Sample Results for Metals in Method 5 and Ambient Air Samples

Results for matrix/matrix spike duplicate analysis of metals in Method 5 and ambient air samples are presented in Table 8-47. All the elements, except for silver, were recovered inside the 100 ±20% accuracy limits specified in the QAPP. Silver was not recovered in the spiked samples or in the spiked blank. A laboratory footnote states that the spiking solution is incompatible with the digestion process. It is not known if the digestion process would affect silver in the field samples in the same manner. Repeatability of these analyses was within the accuracy objectives (RPD ≤20%) for all elements, except for silver. Results for silver were not calculated.

8.8.3 Duplicate Analysis Results for Metals in Method 5 and Ambient Air Samples

Results for duplicate analysis of metals in Method 5 and ambient air samples are presented in Table 8-48. All the results are inside the accuracy objectives (RPD ≤ 20%) specified in the QAPP.

TABLE 8-46. SUMMARY OF REAGENT BLANKS REPORTED ABOVE THE METHOD DETECTION LIMIT FOR METHOD 5 AND AMBIENT AIR SAMPLES ANALYZED FOR METALS

Element	Detection Limit (mg/L)	Analysis Date	Type	Result (mg/L)
Cadmium	0.003	1/06/88	Analytical	0.009
Cadmium	0.003	1/27/88	Analytical	0.007
Cadmium	0.003	1/29/88	Analytical	0.005
Cadmium	0.003	1/29/88	Analytical	0.004
Chromium	0.009	1/29/88	Analytical	0.019
Chromium	0.009	1/29/88	Preparation	0.018
Nickel	0.02	1/06/88	Analytical	0.029
Silver	0.009	1/27/88	Analytical	0.019
Silver	0.009	1/27/88	Analytical	0.012
Silver	0.009	1/29/88	Analytical	0.02
Silver	0.009	1/29/88	Analytical	0.012
Vanadium	0.02	1/06/88	Analytical	0.022
Vanadium	0.02	1/29/88	Analytical	0.022
Zinc	0.006	1/27/88	Analytical	0.008
Zinc	0.006	1/29/88	Analytical	0.007
Zinc	0.006	1/29/88	Preparation	0.014
Lead	0.001	1/26/88	Preparation	0.004
Lead	0.001	12/11/87	HEP Blank	0.004
Lead	0.001	1/27/88	Field Blank M5	1.2
Mercury	0.0002	12/8/87	Preparation	0.0002
Mercury	0.0002	1/27/88	Field Blank M5	0.02
Antimony	0.003	1/27/88	Field Blank M5	0.9
Selenium	0.003	1/27/88	Field Blank M5	1.8

TABLE 8-47. SUMMARY OF MATRIX SPIKE AND MATRIX SPIKE DUPLICATE SAMPLE RECOVERIES FOR METHOD 5 AND AMBIENT AIR SAMPLES ANALYZED FOR METALS

			MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
Analysis Date	Sample Name	Element	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
1/06/88	AM 1103 D	Barium	<11	1170	1200	98	98	0
1/06/88	AM 1103 D	Beryllium	1.3	1100	1200	92	92	0
1/06/88	AM 1103 D	Cadmium	<3.6	1100	1200	92	92	0
1/06/88	AM 1103 D	Chromium	<11	1130	1200	94	94	0
1/06/88	AM 1103 D	Copper	161	1250	1200	91	90	1
1/06/88	AM 1103 D	Nickel	26	1140	1200	93	92	1
1/27/88	AM 1103 D	Silver	<11	215	1200	18[b]	15[b]	18
1/27/88	AM 1103 D	Thallium	<108	1050	1200	88	92	4
1/06/88	AM 1103 D	Vanadium	<24	1180	1200	98	97	1
1/11/88	AM 1103 D	Zinc	69	1210	1200	93	2	
12/16/88	AM 1103 D	Arsenic	<2.4	1150	1200	96	96	0
12/11/88	AM 1103 D	Lead	46	1400	1200	113	108	5
1/06/88	AM 1103 U	Mercury	0.16	2.4	2.4	93	103	10
12/21/87	AM 1103 D	Antimony	<4	1320	1200	110	112	2
12/11/88	AM 1103 D	Selenium	<3.6	1130	1200	94	94	0
1/27/88	AM 1106 D	Barium	<11	1121	1200	93	94	1
1/27/88	AM 1106 D	Beryllium	<1.2	1111	1200	92	93	1
1/27/88	AM 1106 D	Cadmium	<4	1102	1200	92	92	0
1/27/88	AM 1106 D	Chromium	<11	1167	1200	97	97	0
1/27/88	AM 1106 D	Copper	16	1083	1200	89	90	1
1/27/88	AM 1106 D	Nickel	<24	1117	1200	93	94	1
1/27/88	AM 1106 D	Silver	32	233	1200	0[b]	0[b]	NC
1/27/88	AM 1106 D	Thallium	<108	1140	1200	95	98	3
1/27/88	AM 1106 D	Vanadium	<24	1154	1200	96	96	0

(Continued)

TABLE 8-47. (Continued)

			MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
Analysis Date	Sample Name	Element	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
1/27/88	AM 1106 D	Zinc	31	1112	1200	90	92	2
1/26/88	AM 1106 D	Arsenic	<2.4	1152	1200	96	92	4
1/26/88	AM 1106 D	Lead	24	1224	1200	100	98	2
1/18/88	AM 1106 D	Mercury	<0.12	2.28	2.4	95	100	5
1/26/88	AM 1106 D	Antimony	<3.6	1344	1200	112	116	4
1/26/88	AM 1106 D	Selenium	3.6	1248	1200	104	106	2
1/27/88	Blank	Barium		0.92	1	92		
1/27/88	Blank	Beryllium		0.92	1	92		
1/27/88	Blank	Cadmium		0.91	1	91		
1/27/88	Blank	Chromium		0.94	1	94		
1/27/88	Blank	Copper		0.88	1	88		
1/27/88	Blank	Nickel		0.91	1	91		
1/27/88	Blank	Silver		0.2	1	20[b]		
1/27/88	Blank	Thallium		0.96	1	96		
1/27/88	Blank	Vanadium		0.94	1	94		
1/27/88	Blank	Zinc		0.89	1	89		
1/26/88	Blank	Arsenic		0.96	1	96		
1/26/88	Blank	Lead		1.04	1	104		
1/26/88	Blank	Antimony		1416	1200	118		
1/26/88	Blank	Selenium		1.02	1	120		
1/06/88	Blank	Barium		0.96	1	96		
1/06/88	Blank	Beryllium		0.92	1	92		
1/06/88	Blank	Cadmium		0.91	1	91		

(Continued)

TABLE 8-47. (Continued)

			MATRIX SPIKES			MATRIX SPIKE DUPLICATES		
Analysis Date	Sample Name	Element	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery 1 (%)	Recovery 2 (%)	RPD[a] (%)
1/06/88	Blank	Chromium		0.93	1	93		
1/06/88	Blank	Copper		0.91	1	91		
1/06/88	Blank	Nickel		0.95	1	95		
1/27/88	Blank	Silver		0.18	1	18[b]		
1/27/88	Blank	Thallium		0.92	1	92		
1/06/88	Blank	Vanadium		0.95	1	95		
1/11/88	Blank	Zinc		0.94	1	94		
12/16/88	Blank	Arsenic		0.018	0.02	90		
12/11/88	Blank	Lead		1	1	100		
12/21/87	Blank	Antimony		1.18	1	118		
12/11/88	Blank	Selenium		0.9	1	90		

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximium Value - Minimum Value)}}{\text{(Maximum Value + Minimum Value)/2}}$ x 100.

[b] Outside accuracy objective (100±20% recovery) specified in the QAPP.

NC = Not Calculated.

TABLE 8-48. REPLICATE ANALYSIS OF METHOD 5 AND AMBIENT AIR SAMPLES ANALYZED FOR METALS

Sample Name	Date	Element	Result 1 (ug/filter)	Result 2 (ug/filter)	RPD[a] (%)
AM 1103 U	12/08/87	Mercury	2.4	2.64	9.5
AM 1103 U	1/06/88	Barium	1170	1180	1
AM 1103 U	1/06/88	Beryllium	1100	1110	1
AM 1103 U	1/06/88	Cadmium	1100	1100	0
AM 1103 U	1/06/88	Chromium	1130	1130	0
AM 1103 U	1/06/88	Copper	1250	1240	1
AM 1103 U	1/06/88	Nickel	1140	1130	1
AM 1103 U	1/06/88	Vanadium	1180	1160	2
AM 1103 U	1/27/88	Silver	215	183	16
AM 1103 U	1/27/88	Thallium	1050	1110	6
AM 1103 U	1/11/88	Zinc	1210	1180	3
AM 1104 D	12/08/87	Mercury	1.1	0.98	11.5
AM 1103 D	12/11/87	Selenium	1130	1130	0
AM 1103 D	12/11/87	Lead	1400	1340	4
AM 1103 U	12/11/87	Lead	20	23	14
AM 1103 U	12/11/87	Selenium	<3.6	<3.6	NC
AM 1103 D	12/16/87	Arsenic	1150	1150	0
AM 1103 U	12/16/87	Arsenic	<2.4	<2.4	NC
AM 1103 D	12/21/87	Antimony	1320	1340	2
AM 1103 U	12/21/87	Antimony	<4	<4	NC
AM 1103 U	1/06/88	Copper	72	73	1
AM 1103 U	1/06/88	Cadmium	<3.6	13	NC
AM 1103 U	1/06/88	Barium	<11	16	NC
AM 1103 U	1/06/88	Nickel	<24	<24	NC
AM 1103 U	1/06/88	Vanadium	<24	<24	NC
AM 1103 U	1/06/88	Chromium	<11	<11	NC
AM 1103 U	1/06/88	Beryllium	1.3	<1.2	NC
AM 1103 U	1/08/88	Arsenic	<360	<360	NC
AM 1103 U	1/08/88	Arsenic	<72	<72	NC
AM 1103 U	1/11/88	Zinc	59	51	15
AM 1106 D	1/18/88	Mercury	2.28	2.4	5.1
AM 1106 D	1/18/88	Mercury	<0.012	<0.012	NC
AM 1106 D	1/26/88	Selenium	1248	1272	2
AM 1106 D	1/26/88	Arsenic	1152	1104	4
AM 1106 D	1/26/88	Lead	1224	1200	2
AM 1106 U	1/26/88	Lead	11[b]	11[b]	NC
AM 1106 U	1/26/88	Selenium	<3.6	<3.6	NC
AM 1109 D	1/26/88	Arsenic	<2.4	<2.4	NC
AM 1109 U	1/26/88	Arsenic	<2.4	<2.4	NC

(Continued)

TABLE 8-48. (Continued)

Sample Name	Date	Element	Result 1 (ug/filter)	Result 2 (ug/filter)	RPD[a] (%)
AM 1109 U	1/26/88	Lead	19	19	0.0
AM 1103 U	1/27/88	Barium	11	<11	NC
AM 1103 U	1/27/88	Copper	38[b]	34[b]	NC
AM 1103 U	1/27/88	Cadmium	<4	<4	NC
AM 1103 U	1/27/88	Thallium	<108	<108	NC
AM 1103 U	1/27/88	Thallium	<108	<108	NC
AM 1103 U	1/27/88	Silver	<11	28[b]	NC
AM 1103 U	1/27/88	Vanadium	<24	<24	NC
AM 1103 U	1/27/88	Nickel	<24	<24	NC
AM 1103 U	1/27/88	Chromium	<11	38	NC
AM 1103 U	1/27/88	Beryllium	<1.2	<1.2	NC
AM 1103 U	1/27/88	Silver	<11	<11	NC
AM 1103 U	1/27/88	Zinc	19[b]	16[b]	NC
AM 1106 D	1/27/88	Antimony	1344	1392	4
AM 1106 U	1/27/88	Antimony	<3.6	<3.6	NC
AM 1106 D	1/27/88	Barium	1121	1127	1
AM 1106 D	1/27/88	Beryllium	1111	1115	0
AM 1106 D	1/27/88	Cadmium	1102	1112	1
AM 1106 D	1/27/88	Chromium	1167	1159	1
AM 1106 D	1/27/88	Copper	1083	1092	1
AM 1106 D	1/27/88	Nickel	1117	1123	1
AM 1106 D	1/27/88	Silver	233	234	0
AM 1106 D	1/27/88	Thallium	1140	1172	3
AM 1106 D	1/27/88	Vanadium	1154	1158	0
AM 1106 D	1/27/88	Zinc	1112	1139	2
AM 1111 D	1/27/88	Antimony	<3.6	<3.6	NC
M5 Filter Train					
M5S 1106	1/26/88	Arsenic	1.3	1.2	8.0
M5S 1105	1/26/88	Lead	172	172	0
M5P 1103	1/27/88	Selenium	7.1[b]	7.0[b]	NC
M5S 1105	1/28/88	Antimony	15	14	7

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}}$ x 100.

[b] Reported result less than 5 times the detection limit. Values in this range may vary by 50-100%. This value is not used in calculations.

NC = Not Calculated.

8.9 QA/QC Results for Chloride Analyses of Filters

Samples of filters were analyzed for chloride by ion chromatography. Quality control check samples were within the range specified by the QAPP. Laboratory duplicate samples were analyzed to assess measurement precision. These results are presented in Table 8-49.

8.10 QA/QC Results for Spiked Sample Results for Samples Analyzed for Hexavalent Chromium

Samples were spiked in the field with 5 mg/L hexavalent chromium. Table 8-50 presents the results of that analyses. Recovery of the spiked sample was quite good.

8.11 Summary of Spike Samples for Liquid Samples Analyzed for Total Organic Carbon

Two liquid samples were spiked and analyzed for total organic carbon in scrubber effluent and make up water. Results for these analyses are presented in Table 8-51. Recovery of both samples was within 100 ±20% showing good recovery for this matrix.

8.12 QA/QC Results for Solids Characterization

Samples of solid waste feed, and ash were analyzed for proximate and ultimate parameters. Both field duplicate and laboratory duplicate samples were analyzed to assess measurement precision. Precision estimates for the solids characterization analyses are summarized in Table 8-52. These results show measurement variability within expected limits, and with no evidence of excessive imprecision due to analytical technique.

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TABLE 8-49. REPLICATE ANALYSIS OF THE SAMPLES ANALYZED FOR CHLORIDE

Sample Name	Result 1 (mM/L)	Result 2 (mM/L)	RPD[a] (%)
Filter M5P 1103 Imp 2	0.2605	0.261	0.19
Filter M5P 1105 Imp 1	30.76	30.62	0.46
Filter M5P 1106 Imp 2	0.177	0.177	0.00

[a] Relative Percent Difference (RPD) (%) = $\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$

TABLE 8-50. SUMMARY OF SPIKED SAMPLE RESULTS FOR SAMPLES ANALYZED FOR HEXAVALENT CHROMIUM

Sample Name	Parameter	Matrix Spikes Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery (%)
P5	Chromium 6	0	4.4	5	88

TABLE 8-51. SUMMARY OF SPIKED SAMPLE RECOVERIES FOR LIQUID SAMPLES ANALYZED FOR TOTAL ORGANIC CARBON

Sample Name	Parameter	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery (%)
Scrubber Effluent					
1105	TOC	<1	24.2	30	81
Make Up Water					
1105	TOC	<1	24.7	30	82

TABLE 8-52. REPLICATE ANALYSIS OF SOLID SAMPLES ANALYZED FOR PROXIMATE AND ULTIMATE ANALYSES

Sample Name	Parameter	Result 1 (%)	Result 2 (%)	RPD[a] (%)
Dirt				
1105	Dry Loss	11.27	11.42	1
1105	Carbon	2.45	2.36	4
1105	Hydrogen	1.71	1.67	2
1105	Oxygen	12.39	12.94	4
1105	Nitrogen	0.08	0.11	32
1105	Sulfur	<0.01	<0.01	NC
1105	Chlorine	0.1	0.1	0
1105	Ash	83.26	82.82	1
1105	Density[b]	2.33	2.3	1
1105	Heating Value[c]	208	246	17
1110 3	Dry Loss	13.32	13.86	4
1110 3	Carbon	4.23	4.11	3
1110 3	Hydrogen	1.51	1.62	7
1110 3	Oxygen	12.56	13.11	4
1110 3	Nitrogen	0.08	0.08	0
1110 3	Sulfur	0.02	0.02	0
1110 3	Chlorine	0.1	0.1	0
1110 3	Ash	81.69	80.96	1
1110 3	Density[b]	2.25	2.24	0
1110 3	Heating Value[c]	634	609	4
Ash				
1105	Dry Loss	1.54	0.95	47
1105	Carbon	0.5	0.29	53
1105	Hydrogen	0.17	0.18	6
1105	Oxygen	<0.5	<0.5	NC
1105	Nitrogen	<0.02	<0.02	NC
1105	Sulfur	<0.01	<0.01	NC
1105	Chlorine	0.05	0.05	0
1105	Ash	99.34	99.32	0
1105	Density[b]	2.7	2.73	1

(Continued)

TABLE 8-52. (Continued)

Sample Name	Parameter	Result 1 (%)	Result 2 (%)	RPD[a] (%)
1110 3	Dry Loss	1.36	1.04	27
1110 3	Carbon	1.3	1.23	6
1110 3	Hydrogen	0.15	0.18	18
1110 3	Oxygen	1.92	2.39	22
1110 3	Nitrogen	0.02	0.03	40
1110 3	Sulfur	0.04	0.04	0
1110 3	Chlorine	0.04	0.05	22
1110 3	Ash	96.52	96.09	0
1110 3	Density[b]	2.87	2.84	1

[a] Relative Percent Difference (RPD) (%) =

$$\frac{\text{(Maximum Value - Minimum Value)}}{\text{Maximum Value + Minimum Value)/2}} \times 100.$$

[b] Units for density are g/mL.

[c] Units for heating value are BTU/lb.

APPENDIX C

QUALITY ASSURANCE PROJECT PLAN/TEST PLAN

RADIAN
CORPORATION

274-013-03-00

QUALITY ASSURANCE PROJECT PLAN/TEST PLAN
Shirco Portable Pilot Test Unit Demonstration Program at Rose Township/Demode Road Site in Flint, Michigan

23 October 1987

Prepared for:

Mr. Sy Rosenthal
Enviresponse, Inc.
GSA Raritan Depot
Woodbridge Avenue
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Prepared by:

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6.6.1

QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM
for
HWERL Contracts/IAGs/Cooperative Agreements/In-house Projects

Lab Workplan No: 87160Y

Task Start Date: Nov 2, 1987
(for measurement, data gathering, and/or data generation activities)

Project Category: II

QA ID No: SP-210-~~B~~ C gl

Date QAPP Received: 10-26-87

Task Title: Shirco Incinerator Demonstration Test, Rose Township in Flint, Michigan

Technical Project Officer: L. Galer (H. Wall)

Contractor: Enviresponse with Radian as Subcontractor

APPROVALS:

✓ Enviresponse - S. Rosenthal Contractor Project/Task Manager	Signature	10/20/87 Date
viresponse - J. Borris Contractor QA Manager	Signature	10-20-87 Date
✓ Radian - Michael Fuchs Affiliate Task Manager*	Michael Fuchs Signature	10/21/87 Date
✓ Huffman Labs - Dale Raines Other (as appropriate)	Dale Raines Signature	10/22/87 Date
✓ Linda Galer HWERL Technical Project Officer	Linda Galer Signature	10/19/87 Date
✓ Jonn Kingscott HWERL Branch or Staff Chief**	Jol Kingscott Signature	10/19/87 Date
✓ Guy Simes HWERL Quality Assurance Officer	Guy Simes Signature	10-26-87 Date

* Approval signature is required for any ancillary sampling, analytical, or data gathering support provided by a subcontractor or HWERL principal investigator.

** Approval signature from the HWERL Branch or Staff Chief is required for Category I, II, and III extramural projects and for all in-house projects.

WERL (QAPP AF)
(October 1986)

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2	Project Organization and Responsibility	3	1	10-2-87
3	Data Quality Objectives	13	1	10-2-87
4	Sampling Program	72	1	10-2-87
5	Laboratory Procedures/Calibrations/ Quality Control	48	1	10-2-87
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1.0 PROJECT DESCRIPTION

Enviresponse, Inc is under contract to the United States Environmental Protection Agency (USEPA) to provide technical and engineering services in support of various USEPA hazardous waste treatment demonstration programs. One such demonstration program includes testing of a transportable infrared incineration system being operated by Shirco Infrared Systems, Inc. The incinerator is being used at the Rose Township site near Flint, Michigan to treat a selected portion of contaminated soil at the site. Of primary interest are PCBs (polychlorinated biphenyls) and lead, although other organics and metals are also known to be present.

The incinerator system includes a Shirco infrared primary combustor, a secondary combustion chamber, a wet venturi air pollution control system, and a data acquisition and control system. The system is portable and is a candidate technology for the destruction of contaminated materials as part of remedial actions at contaminated sites.

During the test program at the Rose Township site, the unit will first be operated at a single set of conditions for optimum destruction of organic contaminates in the waste. A parametric study will then be conducted to evaluate the effects of temperature and feed rate. Results of the incineration tests will provide the USEPA with information necessary in the evaluation of the Shirco portable infrared incinerator as a technology suitable for the destruction of hazardous constituents in contaminated materials.

Enviresponse, Inc. is serving as the procurement agent of the USEPA during this program. Radian is under contract to Enviresponse, Inc. to provide the sampling and analytical support services necessary to gather the data required to evaluate the suitability of the Shirco system to destroy solid hazardous materials.

The monitoring program will be designed and executed with emphasis on completeness and data quality. A comprehensive quality assurance (QA) and quality control (QC) program will be an integral part of the overall test program. The goal of the QA/QC effort will be to ensure that the data collected are of known precision and accuracy and that they are complete, representative, and comparable.

Figure 1-1 presents the project schedule. Table 1-1 sets out the milestones and their anticipated achievement date.

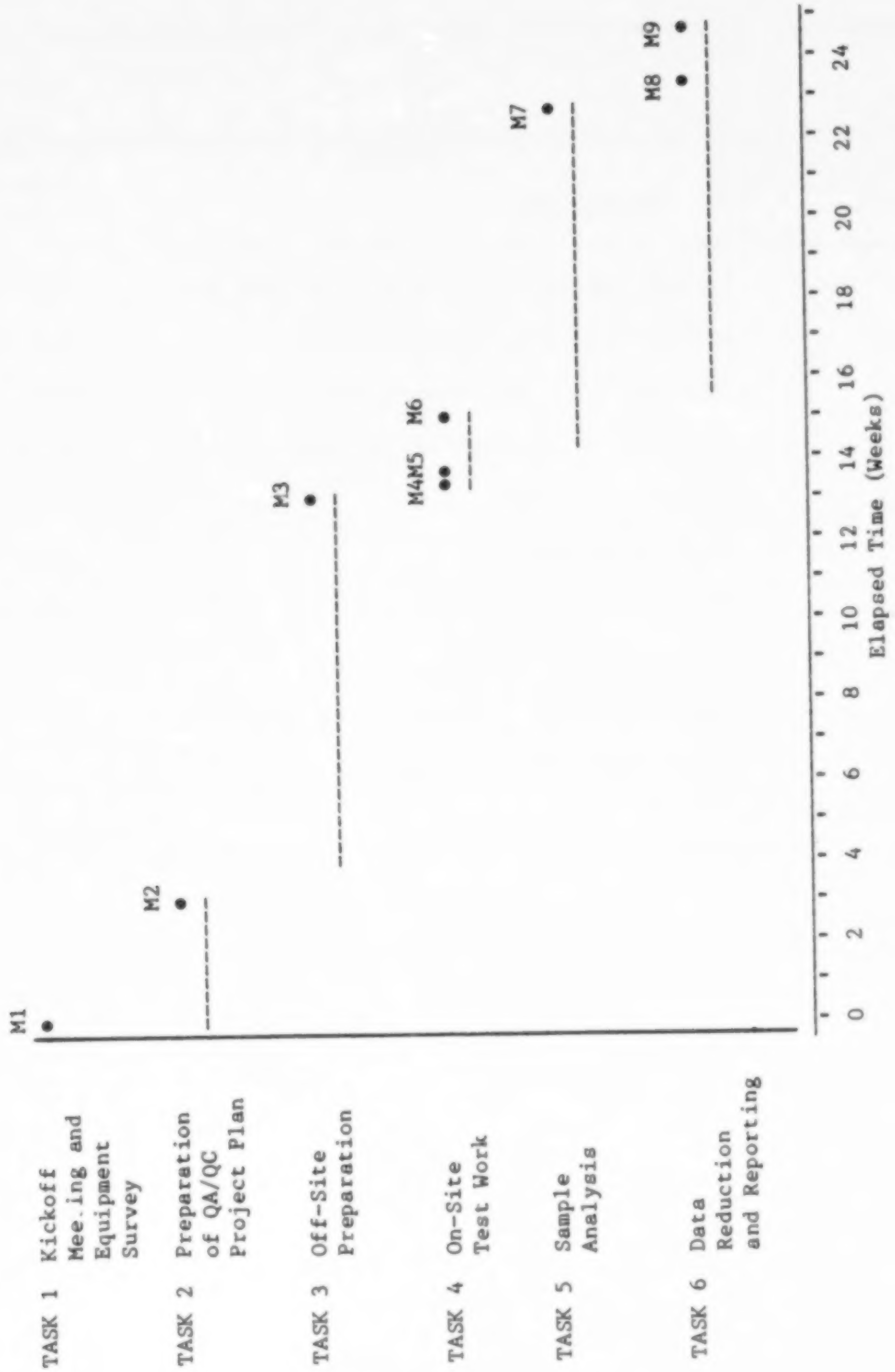


Figure 1-1. Shirco Demonstration Project Schedule

TABLE 1-1. SHIRCO INCINERATOR DEMONSTRATION TEST MILESTONES AND PLANNED DATES *

Milestone	Description	Planned Date
M1	Kickoff Meeting and Equipment Inspection	7/23/87
M2	Submit QA/QC Plan to Enviresponse	10/2/87
M3	Complete Field Test Preparations	10/23/87
M4	Complete CEM Checkout and Equipment Set-Up	10/26/87
M5	Complete Test Burn	10/29/87
M6	Complete Parameterization Study	11/6/87
M7	Complete Analyses	12/30/87
M8	Complete Data Reduction	1/6/88
M9	Submit Draft Final Report to Enviresponse	1/15/88
M10	Submit Final Report to Enviresponse	30 Days after Receipt of Comments

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The project organization for the Shirco Portable Pilot Test Unit sampling program is presented in Figure 2-1. The quality assurance (QA) and quality control (QC) functions have been organized to allow independent review of project activities, while providing on-site QC coordination by the person most knowledgeable of the sampling/analysis activities.

The objectives of the quality control efforts for this program are to assess and to document the precision, accuracy, and adequacy of the process and emission data developed during sampling and analysis.

The QA Coordinator for this project is Mr. D.L. Lewis. While Mr. Lewis coordinates the development and execution of the QA activities for the field testing effort, he will also be responsible for reviewing the QA Project Plan, evaluating the internal QC program, and documenting the results of all QA/QC activities.

As task leader for the sampling effort, Mr. M.J. Krall will be responsible for implementing the project-specific system of quality control activities. He will schedule all on-site QC activities, establish on-site sampling/analysis protocols, and coordinate record keeping and data review and validation.

The analytical task leader, Ms. S.K. Mertens, will be responsible for the off-site analytical effort. Her responsibilities include obtaining valid analytical data for all project samples and adhering to quality control requirements specified in the analytical methods. An outside laboratory has been subcontracted to perform some of the sample analysis during this project.

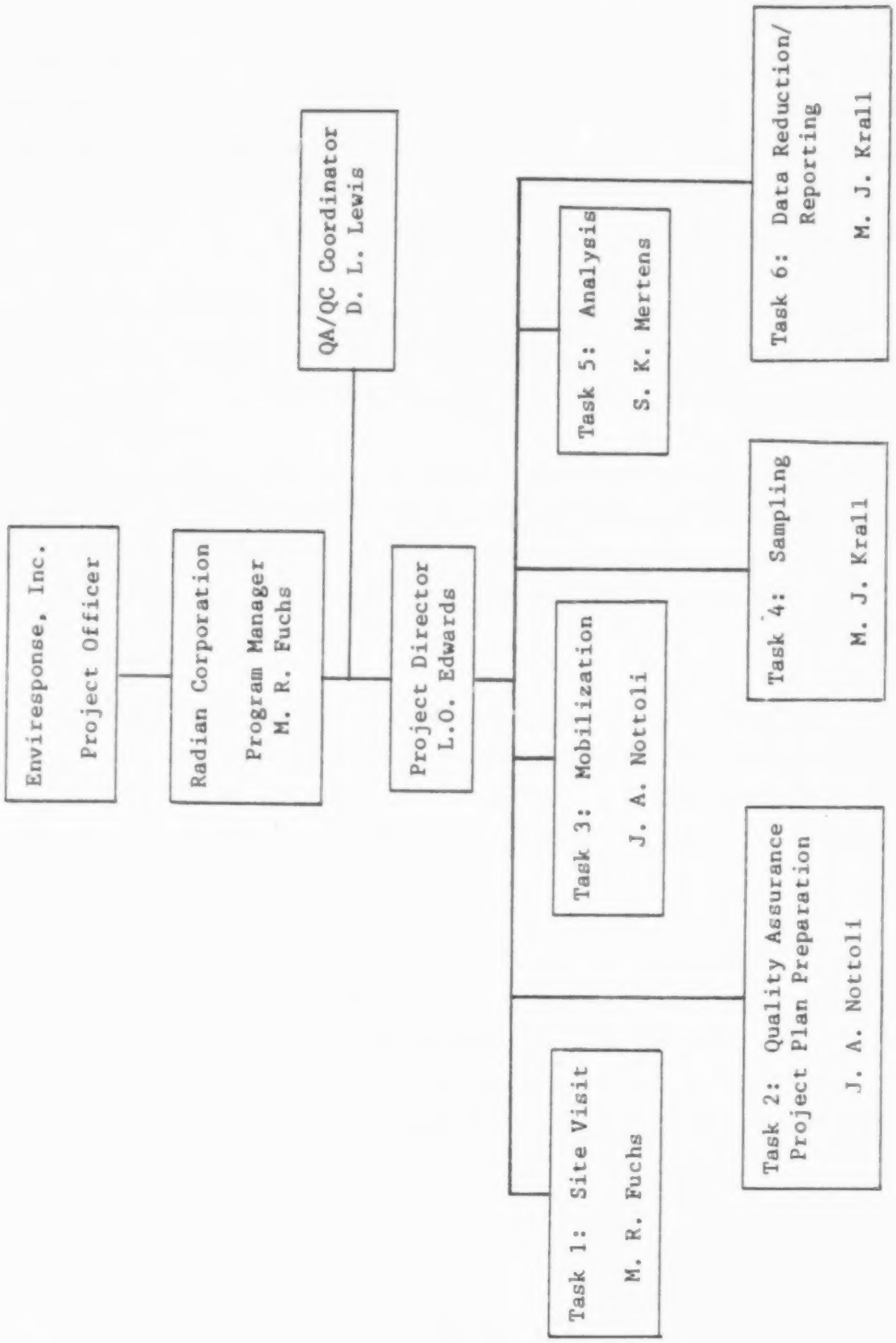

Envirespons e, Inc.
Project Officer
Radian Corporation
Program Manager
M. R. Fuchs
QA/QC Coordinator
D. L. Lewis
Project Director
L.O. Edwards
Task 1: Site Visit
M. R. Fuchs
Task 2: Quality Assurance Project Plan Preparation
J. A. Nottoli
Task 3: Mobilization
J. A. Nottoli
Task 4: Sampling
M. J. Krall
Task 5: Analysis
S. K. Mertens
Task 6: Data Reduction/ Reporting
M. J. Krall

Figure 2-1. Shirco Portable Pilot Test Unit Demonstration Program Organization

Huffman Laboratories, Inc. (Golden, Colorado) will perform chlorine analysis and ash content of the solid waste feed, furnace ash, scrubber solids, and ultimate analysis and higher heating value of the solid waste feed. The remaining analyses will be performed by Radian Corporation in Austin, Texas, and Research Triangle Park, North Carolina.

Mr. D. Raines will coordinate the QA/QC activities during the sample analyses at Huffman Laboratories, Inc. His duties will include reviewing all results of sample analyses, calibrations, blanks, duplicate analyses, and analytical system performance checks. Following receipt of the samples, Mr. Raines will be contacted by phone every other week by Radian to discuss analytical progress and results. He will also submit an analysis and QA/QC report to Radian following the completion of all analytical work.

The Project Director, Mr. L.O. Edwards, is ultimately responsible for the overall technical effort. This responsibility includes the timely, cost-effective execution of all project activities. He will also coordinate preparation of the final report.

The Program Manager, Mr. M.R. Fuchs, has final responsibility for completing this program in accordance with the program objectives and within schedule and budget constraints.

3.0 DATA QUALITY OBJECTIVES

The objectives of the quality assurance plan for this project are two-fold. First, the plan provides the mechanism for ongoing control and evaluation of measurement data quality throughout the course of the project. Second, audit results and quality control data will ultimately be used to define data quality for the various measurement parameters in terms of both precision and accuracy. These data quality estimates will, in turn, be used to define the level of the uncertainty associated with the measured emission concentration values.

All data will be calculated and reported in units consistent with standard EPA and industry practice. Method detection limits (MDLs) are listed in Table 3-1 for organics and Table 3-2 for metals. Table 3-1 lists all organic analytes to be determined in this study. The suitability of any extraction method to be used in conjunction with a specific analyte is given or referenced in Appendix IX (FR 25942-53, July 9, 1987).

Accuracy and precision objectives for analytes are based on results for matrix spikes and matrix spike duplicate (MS/MSD) analyses. The matrix spike compounds and recovery criteria specified in EPA Contract Laboratory Program (CLP) are given for volatile and semivolatile organics as well as metals. Accuracy objectives for dioxins and furans are from SW-846, Method 8280, Third Edition. Precision objectives for analyses are based on matrix spike duplicates. Data quality objectives for the sampling parameters associated with this project are presented in Table 3-3. Data quality objectives for analytical parameters are presented in Table 3-4.

No data quality objectives for total dissolved solids, total suspended solids, ash, ultimate analysis, higher heating value, and pH are presented because these are system parameters to be monitored and are not critical measurements. A full discussion of the quantitative assessment of the measurement system data, particularly precision and accuracy, can be found in Section 9.0.

TABLE 3-1. SUMMARY OF DETECTION LIMITS FOR ORGANICS

Contaminant	Method Detection Limit[a] (ug/L)
Volatiles (Method 8240)	
Acetonitrile	200
Acrolein	500
Acrylonitrile	10
Benzene	2
Bromodichloromethane	2
Bromomethane	2
Carbon disulfide	10
Carbon tetrachloride	2
Chlorobenzene	2
Chlorodibromomethane	2
Chloroethane	2
Chloroform	2
Chloromethane	2
2-Chloro-1,3-butadiene	50
3-Chloropropene	2
1,2-Dibromo-3-chloropropane	2
1,2-Dibromoethane	2
Dibromomethane	2
Dichlorodifluoromethane	2
1,1-Dichloroethane	2
1,2-Dichloroethylene	2
1,1-Dichloroethylene	2
trans-1,2-Dichloroethane	2
trans-1,4-Dichloro-2-butene	50
1,2-Dichloropropane	5
trans-1,3-Dichloropropene	5
cis-1,3-Dichloropropene	5
Ethyl methacrylate	2
Iodomethane	2
Methyl ethyl ketone	10
Methyl methacrylate	2
Methylene chloride	50

(Continued)

TABLE 3-1. (Continued)

Contaminant	Method Detection Limit[a] (ug/L)
Volatiles (Method 8240) (continued)	
Ethylene Oxide	1000
Xylene	10
1,1,1,2-Tetrachloroethane	2
1,1,2,2-Tetrachloroethane	2
Tetrachloroethene	2
Toluene	2
Tribromomethane	2
1,1,1-Trichloroethane	2
1,1,2-Trichloroethane	2
Trichloroethene	2
Trichlorofluoromethane	2
1,2,3-Trichloropropane	50
Vinyl chloride	2
Ethylbenzene	2
Vinyl Acetate	10
Styrene	2
Acetone	50
Methyl isobutyl ketone	10
2-Hexanone	50
Propanenitrile	500
Volatiles (Method 5040)	
Chloromethane	nd
Bromomethane	nd
Vinyl chloride	nd
Chloroethane	nd
Methylene chloride	b
Trichlorofluoromethane	nd
1,1-Dichloroethene	b
1,1-Dichloroethane	b
trans-1,2-Dichloroethene	b
Chloroform	b
1,2-Dichloroethane	b
1,1,1-Trichloroethane	b
Carbon tetrachloride	b
Bromodichloromethane	b

(Continued)

TABLE 3-1. (Continued)

Contaminant	Method Detection Limit[a] (ug/L)
Volatiles (Method 5040) (continued)	
1,2-Dichloropropane	b
trans-1,3-Dichloropropene	b
Trichloroethene	b
Benzene	b
Dibromochloromethane	b
1,1,2-Trichlorethane	b
cis-1,3-Dichloropropene	nd
2-Chlorethylvinyl ether	nd
Bromoform	b
1,1,2,2-Tetrachloroethane	b
Tetrachloroethene	b
Toluene	b
Chlorobenzene	b
Ethyl benzene	b
1,3-Dichlorobenzene	nd
1,2-Dichlorobenzene	nd
1,4-Dichlorobenzene	nd
Semivolatiles (Method 8270)	
Acenaphtalene	2
Acenaphthene	10
Acetophenone	10
2-Acetylaminofluorene	1000
4-Aminobiphenyl	200
Aniline	20
Anthracene	10
Aramite	c
Benzo(a)anthracene	10
Benzenethiol	c
Benzidine	1000
Benzo(a)pyrene	10
Benzo(b)fluoranthrene	10
Benzo(ghi)perylene	10
Benzo(k)fluoranthrene	10
p-Benzoquinone	1000
Benzoic acid	500

(Continued)

TABLE 3-1. (Continued)

Contaminant	Method Detection Limit[a] (ug/L)
Semivolatiles (Method 8270) (continued)	
Benzyl alcohol	50
2-Chloroethyl vinyl ether	1000
4-Chlorophenyl phenyl ether	50
Dibenzofuran	2
Dibenz(a,h)pyrene	10
Bis(2-chloroethoxy)ethane	10
Bis(2-chloroethyl)ether	10
Bis(2-chloroisopropyl)ether	10
Bis(2-ethylhexyl)phthalate	10
4-Bromophenyl phenyl ether	10
Butylbenzylphthalate	10
2-sec-Butyl-4,6-dinitrophenol	c
p-Chloroaniline	100
Chlorobenzilate	c
p-Chloro-m-cresol	10
2-Chloronaphthalene	10
2-Chlorophenol	10
3-Chloropropionitrile	c
Chrysene	10
ortho-Cresol	10
para-Cresol	10
Dibenz(a,h)anthracene	10
Dibenzo(a,e)pyrene	c
Dibenzo(a,i)pyrene	c
m-Dichlorobenzene	10
o-Dichlorobenzene	10
p-Dichlorobenzene	10
3,3'-Dichlorobenzidine	20
2,4-Dichlorophenol	10
2,6-Dichlorophenol	10
Diethylphthalate	10
3,3'-Dimethoxybenzidine	10000
p-Dimethylaminoazobenzene	200
3,3'-Dimethylbenzidine	10000
2,4-Dimethylphenol	10
Dimethylphthalate	10
Di-n-butylphthalate	10
1,2-Dichlorobenzene	2

(Continued)

TABLE 3-1. (Continued)

Contaminant	Method Detection Limit[a] (ug/L)
Semivolatiles (Method 8270) (continued)	
1,3-Dichlorobenzene	2
1,4-Dichlorobenzene	2
7,12-dimethylbenz(a)anthracene	5
2,2-Dimethylphenethyamine	c
Isophorone	c
Methyl methanesulfonate	20
2-methylnaphthalene	2
1,4-Dinitrobenzene	100
4,6-Dinitro-o-cresol	50
2,4-Dinitrophenol	50
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
Di-n-octylphthalate	10
Di-n-propylnitrosoamine	20
Diphenylamine/Diphenylnitrosamine	10
1,2-Diphenylhydrazine	10
Fluoranthene	10
Fluorene	10
Hexachlorobenzene	10
Hexachlorobutadiene	10
Hexachlorocyclopentadiene	10
Hexachloroethane	10
Hexachlorophene	20000
Hexachloropropene	10
Ideno(1,2,3-cd)pyrene	10
Isosafrole	100
Methapyrilene	c
3-Methylcholanthrene	100
4,4'-Methylenebis(2-chloroaniline)	200
Naphthalene	10
1,4-Naphthoquinone	100
1-Naphthylamine	100
2-Naphthylamine	100
p-Nitroaniline	50
4-Nitrophenol	10
N-Nitrosodi-n-butylamine	50
N-Nitrosodiethylamine	100
N-Nitrosodimethylamine	100
N-Nitrosomethylethylamine	100

(Continued)

TABLE 3-1. (Continued)

Contaminant	Method Detection Limit[a] (ug/L)
Semivolatiles (Method 8270) (continued)	
Pyridine	20
N-Nitrosodiphenylamine	10
2-Nitrophenol	10
4-Nitroaniline	10
3-Nitroaniline	10
2-Nitroaniline	10
N-Nitrosomorpholine	200
N-Nitrosopiperidine	200
N-Nitrosopyrrolidine	200
5-Nitro-o-toluidine	200
Pentachlorobenzene	10
Pentachloroethane	10
Pentachloronitrobenzene	100
Pentachlorophenol	50
Phencetin	100
Phenanthrene	10
Phenol	10
2-Picoline	100
Pronamide	100
Pyrene	10
Resorcinol	1000
Safrole	100
1,2,4,5-Tetrachlorobenzene	10
2,3,4,6-Tetrachlorophenol	100
1,2,4-Trichlorobenzene	10
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	10
Tris(2,3-dibromopropyl)phosphate	nd
PCBs (Method 680)	
Monochlorobiphenyls	0.02
Dichlorobiphenyls	0.02
Trichlorobiphenyls	0.02
Tetrachlorobiphenyls	0.02
Pentachlorobiphenyls	0.02
Hexachlorobiphenyls	0.02
Heptachlorobiphenyls	0.02
Octachlorobiphenyls	0.02
Nonachlorobiphenyls	0.02
Decachlorobiphenyls	0.02

(Continued)

TABLE 3-1. (Continued)

Contaminant	Method Detection Limit[a] (ug/L)
Dioxins and Furans (Method 8280)	
Hexachlorodibenzo-p-dioxins	0.01
Hexachlorodibenzofuran	0.01
Pentachlorodibenzo-p-dioxins	0.01
Pentachlorodibenzofuran	0.01
Tetrachlorodibenzo-p-dioxins	0.01
Tetrachlorodibenzofuran	0.01
2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.01
Heptachlorodibenzo-p-dioxin	0.01
Heptachlorodibenzofuran	0.01
Octachlorodibenzo-p-dioxin	0.01
Octachlorodibenzofuran	0.01

[a] MDL's are given for groundwater. Practical quantitation limits for feed, ash and gas samples are dependent upon the matrix and sampling conditions and may be higher than the MDL's given by a factor of 10-10,000. Actual MDL's used will be included in the final report.

[b] Method 5040 does not introduce an aqueous phase. The detection limit of any particular analyte is generally set at 25 ng. This would correspond for benzene, for example, to a gaseous concentration of 0.4 ppbv. Detection limits for other analytes would be inversely proportional to their relative molecular weight.

[c] Detection limit not given, standard unavailable.

nd - Not determined.

TABLE 3-2. SUMMARY OF METHOD DETECTION LIMITS FOR METALS

Analyte	Analysis Type	Method Detection Limit (mg/L)
Silver	ICPES	0.009
Arsenic	GFASS	0.003
Barium	ICPES	0.009
Beryllium	ICPES	0.001
Cadmium	ICPES	0.003
Chromium	ICPES	0.010
Copper	ICPES	0.010
Mercury	CVAAS	0.0002
Nickel	ICPES	0.020
Lead	ICPES	0.050
Antimony	ICPES	0.06
Selenium	GFAAS	0.005
Thallium	GFAAS	0.003
Vanadium	ICPES	0.020
Zinc	ICPES	0.006

ICPES - Inductively Coupled Plasma Spectroscopy.
CVAAS - Cold Vapor Atomic Absorption Spectroscopy.
GFAAS - Graphite Furnace Atomic Absorption Spectroscopy.

TABLE 3-3. SUMMARY OF ESTIMATED PRECISION, ACCURACY, AND DATA CAPTURE OBJECTIVES

Parameter (Method)	Precision	Accuracy	Data Capture[a]
PCDD/PCDF (Flue Gas - SASS and Solid/Liquid)	50%[b]	± 50%[c]	90%
PCBs (Flue Gas - SASS, Ambient and Solid/Liquid)	50%[b]	± 50%[c]	90%
Volatile Organics (Flue Gas - VOST and Solid/Liquid)	30%[b]	± 50%[c]	90%
Semi-Volatile Organics (Flue Gas - SASS and Solid/Liquid)	30%[b]	± 50%[c,g]	90%
Particulate Matter (Flue Gas - Method 5)	11%[d]	± 10%[d]	90%
HCl (Flue Gas - Caustic Impinger)	10%[b]	± 10%[c]	90%
Flue Gas - Velocity/Volumetric Flow Rate (Methods 1 and 2)	6%[d]	± 10%[d]	95%
Flue Gas - Fixed Gases/Molecular Weight (Method 3)	10%[f]	± 20%[e]	90%
Flue Gas - Moisture (Method 4)	20%[d]	± 10%[d]	90%
Flue Gas O_2 (Continuous Emission Monitoring System)	20%[f]	± 20%[e]	90%
Flue Gas NO_x (Continuous Emission Monitoring System)	20%[f]	± 20%[e]	90%
Flue Gas CO (Continuous Emission Monitoring System)	20%[f]	± 20%[e]	90%
Flue Gas CO_2 (Continuous Emission Monitoring System)	20%[f]	± 20%[e]	90%
Flue Gas THC (Continuous Emission Monitoring System)	20%[f]	± 25%[e]	90%

(Continued)

TABLE 3-3. (Continued)

Parameter (Method)	Precision	Accuracy	Data Capture[a]
Chlorine (Solid/Liquid)	20%[b]	± 10%[c]	90%
EP Toxicity Metals (Solid/Liquid)	20%[b]	± 25%[e]	90%
TCLP (Solid/Liquid)	30%[b]	± 50%	90%
Metals (Flue Gas and Solid/Liquid)	20%[b]	± 15%[c]	90%
Wind Direction	N/A	± 3°	90%
Wind Speed	N/A	± 0.5 mph	90%
Temperature	± 1°C	± 1°C	90%

[a] Valid data percentage of total tests conducted

[b] Relative percent difference for duplicate analysis, where

$$\text{RPD} = \frac{\text{Maximum Value} - \text{Minimum Value}}{(\text{Maximum Value} + \text{Minimum Value})/2} \times 100\%$$

[c] Relative error (%) based on recovery of matrix spike analytes native to each analytical method matrix, where:

$$\% = \frac{\text{Measured Value} - \text{Spiked Value}}{\text{Spiked Value}} \times 100\%$$

[d] Precision and accuracy estimate based on results of EPA collaborative tests

[e] Relative error (%) for audit analyses, where:

$$\% = \frac{\text{Measured Value} - \text{Theoretical Value}}{\text{Theoretical Value}} \times 100\%$$

[f] Coefficient of variation (CV) for daily analyses of control sample prior to and after daily testing, where:

$$\text{CV} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100\%$$

[g] Phenols accuracy may fall outside this range; see Table 3-4.

TABLE 3-4. PRECISION, ACCURACY, AND COMPLETENESS OBJECTIVES FOR ANALYSES OF VOLATILE AND SEMIVOLATILE ORGANIC ANALYSES

Critical Measurement	Precision[b] (%)	Accuracy[c] (%)	Completeness[d] (%)
Volatile Organics (SW-846 Method 8240)[a]			
1,1-Dichloroethene	20	61-145	95
Trichloroethene	20	71-120	95
Chlorobenzene	20	75-130	95
Toluene	20	76-125	95
Benzene	20	76-127	95
Semivolatile Organics (SW-846 Method 8270)[a]			
1,2,4-Trichlorobenzene	20	39-98	95
Acenaphthene	20	46-118	95
2,4-Dinitrotoluene	20	24-96	95
Pyrene	20	26-127	95
N-Nitroso-di-n-propylamine	20	41-116	95
1,4-Dichlorobenzene	20	36-97	95
Pentachlorophenol	20	9-103	95
Phenol	20	12-89	95
2-Chlorophenol	20	27-123	95
4-Chloro-3-methylphenol	20	23-97	95
4-Nitrophenol	20	10-80	95
Metals (Methods 6010 and 7000 series)	20	75-125	95
PCBs (EPA Method 680)	40	50-125	95
Dioxins and Furans (SW-846 Method 8280)			
Hexachlorodizenzo-p-dioxins	20	72-92	95
Hexachlorodibenzofuran	20	61-104	95
Pentachlorodibenzo-p-dioxins	20	65-93	95
Pentachlorodibenzofuran	20	70-99	95
Tetrachlorodibenzo-p-dioxins	20	46-100	95
Tetrachlorodibenzofuran	20	57-100	95
2,3,7,8-Tetrachlorodibenzo-p-dioxin	20	76-104	95

[a] Accuracy objectives based on matrix spike recovery limits specified in EPA CLP Statement of Work for Organic Analyses, Sept. 1986.

[b] Difference between matrix spike duplicate recoveries. The RPD for sample matrix spike duplicate analyses must be $\leq 20\%$ for analyte concentrations greater than 200 ppb; 100% for analyte concentrations less than 200 ppb.

[c] Percent recovery for matrix spikes.

[d] Valid data, expressed as a percentage of possible measurement data.

The QA/QC program focus will be upon controlling measurement error within these estimated limits of measurement uncertainty. It should be noted that these are estimates which are, in most cases, described in the methods and represent the range of results which can be expected from these methods based on actual field sampling results and laboratory-based QA/QC studies. It is therefore reasonable to expect that the measurement errors associated with this project will be within the estimates shown on Table 3-3.

In the event that ongoing QA/QC procedures reveal that a measurement's error has exceeded these estimated limits, the source of the excessive error will be immediately identified and corrective action taken, as described in Section 10. If data fall outside the acceptable range of precision and accuracy, even after corrective action has been taken, those data points will be flagged in the final reports the precision and accuracy for the measurement will be reported as determined using the actual data. Additionally, if possible, alternative procedures (either sampling or analytical) may be considered and recommended to the client. Any changes or additions would necessarily be agreed to by all parties before implementation.

4.0 SAMPLING PROGRAM

This section provides descriptions of the incineration process, the test program, sampling locations, sampling procedures, and analytical procedures that will be used during the Shirco incineration demonstration test.

4.1 Process Description

Solid waste processed at the Demode Road site will be incinerated in the Shirco Portable Pilot Test Unit designed and manufactured by Shirco Infrared Systems, Inc. of Dallas, Texas. The system consists of a feed metering system, infrared primary chamber, supplemental propane-fired secondary chamber, venturi scrubber, data collection and control systems, and heating element power centers, all enclosed in two 45 foot trailers.

4.1.1 General Process Description

Up to 150 lbs/hr of waste material can be fed to a hopper mounted over a metering conveyor belt; during this study, feed rates are expected to range between 35-90 lbs/hr. The conveyor is shrouded and equipped with rubber skirts to minimize infiltration of air or escape of furnace gases. An adjustable guillotine-type gate is provided at the conveyor discharge. The gate distributes the material across the width of the metering belt and assists in furnace sealing. Final feed area sealing is provided by an additional adjustable knife gate in the feed chute into the furnace. The metering belt is synchronized with the furnace conveyor to control the material feed rate.

The incinerator conveyor moves the waste material through the insulated heating modules (primary unit) where it is brought to a combustion temperature around 1850°F by infrared heating elements. Rotary rakes gently turn the material to ensure adequate mixing and complete burnout. When the ash reaches the discharge end of the furnace, it drops off the belt through a chute and into an enclosed hopper. Combustion air is supplied to the primary unit through a series of overfire air ports at various points along the length of the chamber. The gas flow in the incinerator is countercurrent to the conveyed waste feed material.

Exhaust gas exits the primary chamber near the feed module into a secondary chamber (afterburner) where a propane-fired burner combusts any residual organics present in the exhaust gas at a design temperature of 2300°F. The secondary burners are set to burn at a predetermined temperature. Secondary air is supplied to the afterburner to ensure adequate excess oxygen levels for complete combustion. Exhaust gas from the secondary incinerating unit is then quenched by a water-fed venturi scrubber to remove particulate matter and acid gases. The gas is then transferred to the exhaust stack by an induced draft fan and discharged to the atmosphere.

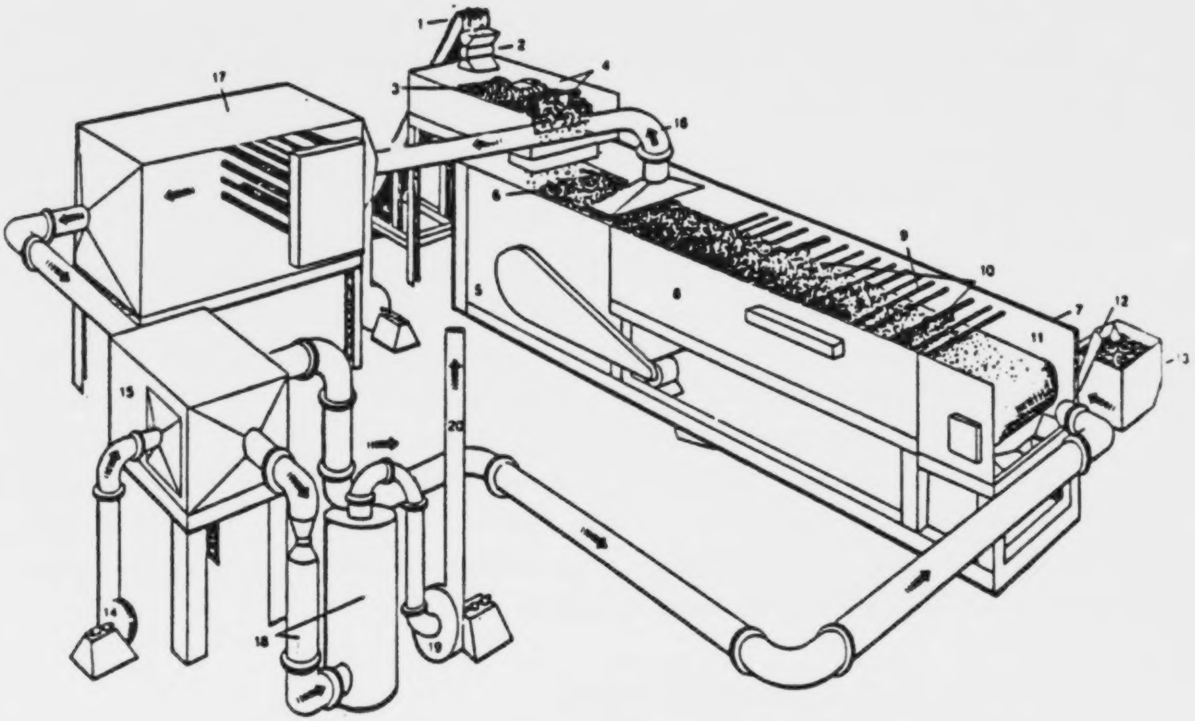
4.1.2 Detailed Description of the Incinerating Unit

A detailed description of the incinerating unit is presented below. A layout diagram illustrating the various subunits of the incinerator and the sampling locations is in Figure 4-1.

4.1.2.1 Incinerating Unit Description

The Portable Pilot Test Unit consists of the following major mechanical subunits and components:

- Waste Feed System,
- Primary Incineration Chamber,
- Secondary Combustion Chamber,
- Emissions Control System, and
- Exhaust System.



Sampling points include:

- Feed solids (1)
- Ash solids (13)
- Primary combustion offgases (16)
- Stack flue gas (20)

Not depicted are the scrubber-related sampling points including make-up water (in holding tank), scrubber water (from clarifier) and scrubber solids (from clarifier).

Figure 4-1. Illustration of the Shirco Incinerator. The main features are the primary combustion chamber (8), the secondary process chamber (17), and the venturi scrubber (18).

These subunits will be transported to the Demode Road site and assembled into one unit. Periodic inspections and maintenance of all mechanical units are accomplished on site and during installation and disassembly. A description of each subunit follows.

4.1.2.2 Waste Feed System

Waste material is fed to a hopper mounted over a metering conveyor belt. The metering belt is synchronized with the furnace conveyor to control the material feed rate. The thermal processor conveyor moves the waste material through the insulated heating modules where it is brought to combustion temperature by infrared heating elements. Rotary rakes gently turn the material to ensure adequate mixing and complete burnout. When the residual material reaches the discharge end of the furnace, it drops off the belt through a chute and into an enclosed hopper. The material is then discharged by means of a screw conveyor to a sealed 55 gallon drum.

4.1.2.3 Primary Incineration Chamber

The primary chamber consists of a rectangular cross-section box constructed of 1/4 inch A36 carbon steel insulated with layers of ceramic fiber blanket insulation and mounted on stainless steel studs retained with ceramic fasteners. The insulation is manufactured by Carborundum Co. and consists of an 85% silica/15% alumina asbestos-free blend which can sustain a continuous surface temperature of 2400°F. The insulation is 7 inches thick on the chamber top, sides, and end plates and 5-inches on the chamber bottom.

The waste material is conveyed through the furnace on a woven wire belt which is supported on high-temperature alloy shafts. The shafts are supported by external flangemount bearings. A friction drive system is used to pull the belt through the furnace. The belt is woven from 16 gauge, 314 stainless steel to a thickness of 0.25-inch. The belt speed is controlled via constant speed motor/variable speed reducer and can be adjusted to achieve waste material residence times of 6 to 60 minutes.

Infrared energy is provided by 18 transversely-mounted silicon carbide heating elements mounted on 3.07-inch centers at a height of 8.75 inches above the furnace belt. These 1.0-inch OD heating elements are manufactured by Carborundum Co. and are rated at 33.1 watts/in^2 of radiating surface with a maximum surface temperature of 2000°F. The primary chamber is divided into two temperature control zones with nine heating elements serving each zone. The temperature in each zone can be precisely controlled from 500 to 1850°F.

Four rotary rakes are used to gently turn the material on the belt to increase exposure. The rotary rakes consist of "L" shaped fingers (0.19-inch OD Inconel 601) welded to 316 stainless steel shafts which rotate at a rate of 1.8 revolutions per inch of belt travel. The shafts are synchronized with the belt speed through a chain-and-sprocket drive system.

Combustion air is supplied to the primary chamber through a series of sixteen 1.0-inch diameter overfire air ports located 1.75 inches above the belt surface. Combustion air flow rate and distribution to each group of four ports (two groups per chamber side) are controlled by manual blast gate valves. The combustion air fan is manufactured by New York Blower Co. and is rated at 185 scfm at a static pressure of 0.25 inches water column. The combustion air supply can be controlled to provide both oxidizing or reducing atmospheres.

4.1.2.4 Secondary Combustion Chamber

The Secondary Combustion Chamber is constructed and insulated in manner very similar to the Primary Incineration Chamber. Its purpose is to thermally destroy the combustible off-gas compounds carried in the exhaust gas from the Primary Incineration Chamber. The internal cross-sectional area of the chamber is 1.56 ft^2 and the internal volume is 28.9 ft^3. The unit is designed for 0-200% excess air operation with a maximum continuous operating temperature of 2300°F and a minimum residence time of 4.3 seconds at maximum actual flow conditions.

Supplemental fuel firing is supplied by a 375,000 Btu/hr forced air nozzle mixed burner. The burner is equipped with a continuous pilot flame monitor, automatic fuel shut-off and purge system interlocked with secondary chamber temperature, and manual fuel control valve. Combustion air supply to the burner is controlled by manually adjustable air registers. Auxiliary fuel firing is supplied by a 36,700 Btu/hr, forced air, premix gas burner.

Secondary combustion air is supplied to the afterburner through the two 1.0-inch diameter ports located at the top of the chamber, directed at the interface of the burner flame pattern and the exhaust gas inlet flow. Secondary combustion air is supplied by the same fan used to supply the primary chamber combustion air. A splitter manifold with dampers at the fan outlet allows air distribution to both chambers. Adjustment of air flow rate to the secondary chamber is by means of a manual blast gate valve.

4.1.2.5 Emissions Control System

The emissions control system consists of a venturi scrubber and droplet separator tower. Water is used as the scrubbing liquor. The venturi section and separator tower are each equipped with two water spray nozzles. The maximum water flow rate is 2 gpm to the venturi and 10 gpm to the separator tower. The venturi section is equipped with a manually adjustable plumb bob to control gas phase pressure drop between 8 and 14 inches of water and effect maximum collection efficiency.

The scrubber is equipped with an integral 45 gallon sump tank, external 50 gallon holding tank, and recirculation pump for effluent handling and disposal. Makeup water is supplied as required by a makeup water pump to account for evaporative losses. Total scrubber water blowdown will be approximately 30 to 45 gallons per day.

In addition to removing particulate matter, the scrubber cools the gases from their incoming temperature (1000°F to 2300°F). The scrubbed gas is drawn from the venturi scrubber by an induced draft fan located downstream of the scrubber.

4.1.2.6 Exhaust System

An induced draft fan, rated at 400 acfm at static pressure of 14 inches of water, exhausts the scrubbed gas through the stack. Exhaust gas is then vented to the atmosphere through a 4.0-inch ID stack which extends through the roof of the trailer to an elevation of 23.6 feet above grade (10'-0" above trailer roof). The stack is equipped with ports to accommodate sampling. Access to the sampling ports is from the trailer roof.

4.2 Test Program

The Rose Township Shirco Portable Pilot Test Unit demonstration program will be conducted in two stages; see Table 4-1. During the Superfund Innovative Technology Evaluation (SITE) test burn (days 2, 3, 4), the unit will operate at a set of normal or optimum conditions. Measurements will be made in triplicate (i.e., single testing of each stream on 3 separate days) at each sampling location. Table 4-2 presents a summary of the SITE test burn program including sampling frequencies, sampling methods, and analytical parameters and methods for each sampling location.

In the subsequent continuous run (days 5 through 12), parametric factors affecting incinerator performance are to be evaluated. During the continuous run, a subset of parameters measured in the SITE test burn will be monitored. Table 4-3 presents the sampling and analysis summary for that stage of the program.

TABLE 4-1. PROPOSED TEST MATRIX

Day	Run	Time	Operating Temperature (°F) Furnace	SCC	Approximate Waste Feed (lbs/hr)	Furnace Residence Time (min)	Atmosphere
1	0[a]	0900-1800	1600	2200	40-60	20	Oxidizing
2	1	0900-1800	1600	2200	40-60	20	Oxidizing
3	2	0900-1800	1600	2200	40-60	20	Oxidizing
4	3	0900-1800	1600	2200	40-60	20	Oxidizing
5	4	0900-1300	1600	1800[b]	35-50	25	Oxidizing
5	5	1400-1800	1600	2200	40-60	20	Oxidizing
6					Downtime		
7					Downtime		
8	6	0900-1300	1600	1800	50-75[c]	15	Oxidizing
8	7	1400-1530	1600	2200	60-90	10	Oxidizing
8	8	1600-1800	1600	1800	50-75[c]	15	Non-Oxidizing
9	9	0900-1300	1200	2200	35-50	25	Non-Oxidizing
9	10	1400-1800	1200	1800	35-50	25	Oxidizing
10	11	0900-1300	1200	2200	40-60	20	Non-Oxidizing
10	12	1400-1800	1200	1800	40-60	20	Oxidizing
11	13	0900-1300	1200	2200	50-75	15	Non-Oxidizing
11	14	1330-1530	1200	1800[b]	50-75	15	Oxidizing
11	15	1600-1800	1200	1800	50-75[c]	15	Non-Oxidizing
12	16	0900-1300	900	2200	35-50	25	Non-Oxidizing
12	17	1330-1530	900	1800	40-60	20	Non-Oxidizing
12	18	1600-1800	900	1800	40-60[c]	25	Non-Oxidizing

[a] Formal sampling not proposed. System shakedown and checkout to be performed.

[b] Secondary combustion chamber to be heated electrically.

[c] Feed to be blended with 3 to 5% fuel oil.

TABLE 4-2. SAMPLING AND ANALYSIS SUMMARY FOR ROSE TOWNSHIP-DEMODE ROAD SITE TEST BURN

Source	Sample Collection Frequency	Sampling Method	Analysis Parameters	Analysis Method
Stack Gas	Composite over 1-3 hour period	EPA Method 5 with 0.1 N NaOH	Particulate Matter HCl Volumetric flowrate Moisture Metals (particulate on filter)	EPA Method 5 Ion Chromatography EPA Methods 1-4 EPA Method 4 SW 6010, 7060, 7041, 7421, 7740, 7470/7471
	Composite over 3-6 hour period	SASS (SW 0020)	PCB	EPA 680
	Composite over 3-6 hour period	SASS (SW 0020)	Semi-Volatile Priority Pollutants (plus 10 highest peaks) PCDD/PCDF	SW 8270 SW 8280
	Composite over 4 hour period	Modified Method 5 (No filter/0.1 N NaOH)	Soluble Chromium	M218.4
		Gas Bag (Grab)	CO_2, O_2	EPA Method 3
	6 pairs of samples over 2 hour period	VOST (SW 0030)	Volatile Priority Pollutants (plus 10 highest peaks)	SW 5040
	Continuous	Continuous emission monitors	O_2 CO_2 CO THC NO_x	Paramagnetic NDIR NDIR FID Chemiluminescence
Primary Furnace Offgas	Composite over 1-3 hour period	EPA Method 5 with 0.1 N NaOH	Particulate Matter HCl Volumetric flowrate Moisture Metals (particulate on filter)	EPA Method 5 Ion Chromatography EPA Methods 1-4 EPA Method 4 SW 6010, 7060, 7041, 7421, 7740, 7470/7471
	Composite over 3-6 hour period	SASS (SW 0020)	PCB	EPA 680
	Composite over 3-6 hour period	SASS (SW 0020)	Semi-Volatile Priority Pollutants (plus 10 highest peaks) PCDD/PCDF	SW 8270 SW 8280

(Continued)

TABLE 4-2. (Continued)

Sourc	Sample Collection Frequency	Sampling Method	Analysis Parameters	Analysis Method
	Composite over 4 hour period	Modified Method 5 (No filter/0.1 N NaOH)	Soluble Chromium	M218.4
		Gas Bag (Grab)	CO_2, O_2	EPA Method 3
	6 pairs of samples over 2 hour period	VOST (SW 0030)	Volatile Priority Pollutants (plus 10 highest peaks)	SW 5040
	Continuous	Continuous emission monitors	O_2	Paramagnetic
			CO_2	NDIR
			CO	NDIR
			THC	FID
			NO_x	Chemiluminescence
Solid Waste Feed	Grab sample every 30 minutes and composite	Scoop or Trowel (S007)	Chlorine	A003
			PCB	EPA 680
			PCDD/PCDF	SW 8280
			Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
			TCLP	SW 8270, 8240, 6010, 7060, 7041, 7421
			EP Toxicity	SW 1310
			Volatile Priority Pollutants (plus 10 highest peaks)	SW 8240
			Semi-volatile Priority Pollutants (plus 10 highest peaks)	SW 8270
			Moisture, Ash	A001
			Higher Heating Value	A006
			Ultimate	A003
			Density	GRAV/VOL
Furnace Ash	Sample composite at end of test period	Trier (S006)	PCB	EPA 680
			PCDD/PCDF	SW 8280
			Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
			EP Toxicity	SW 1310
			TCLP	SW 8270, 8240, 6010, 7060, 7041, 7421
			Volatile Priority Pollutants (plus 10 highest peaks)	SW 8240
			Semi-volatile Priority Pollutants (plus 10 highest peaks)	SW 8270
			Moisture, Ash	A001
			Chlorine	A003
			Density	GRAV/VOL

(Continued)

TABLE 4-2. (Continued)

Source	Sample Collection Frequency	Sampling Method	Analysis Parameters	Analysis Method
Scrubber Water	Sample at end of test period	Dipper (S002)	PCB	EPA 680
			PCDD/PCDF	SW 8280
			pH	SW 9040
			Chloride	Ion Chromatography
			Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
			EP Toxicity	SW 1310
			TCLP	SW 8270, 8240, 6010, 7060, 7041, 7421
			Volatile Priority Pollutants (plus 10 highest peaks)	SW 8240
			Semi-volatile Priority Pollutants (plus 10 highest peaks)	SW 8270
			Total Organic Carbon	M415.1
			Total Suspended Solids	M160.1
			Total Dissolved Solids	M160.2
Scrubber Solids	Sample at end of test period composite	Dipper (S002)	PCB	EPA 680
			PCDD/PCDF	SW 8280
			Chlorine	A003
			Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
			EP Toxicity	SW 1310
			TCLP	SW 8270, 8240, 6010, 7060, 7041, 7421
			Volatile Priority Pollutants (plus 10 highest peaks)	SW 8240
			Semi-volatile Priority Pollutants (plus 10 highest peaks)	SW 8270
			Ash	A003
			Ultimate	A003
			Density	GRAV/VOL
Scrubber Make-Up Water	Sample at end of test period	Tap (S004)	PCB	EPA 680
			PCDD/PCDF	SW 8280
			pH	SW 9040
			Chloride	Ion Chromatography
			Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
			Volatile Priority Pollutants (plus 10 highest peaks)	SW 8240
			Semi-volatile Priority Pollutants (plus 10 highest peaks)	SW 8270
			Total Organic Carbon	M415.1
			Total Suspended Solids	M160.1
			Total Dissolved Solids	M160.2

(Continued)

TABLE 4-2. (Continued)

Source	Sample Collection Frequency	Sampling Method	Analysis Parameters	Analysis Method
Ambient Air	Continuous over 24-hour period; one upwind and one downwind	PUF	PCB	EPA 680
	Continuous over 24-hour period; one upwind and one downwind	Hi-Volume Sampler	Metals	SW 6010, 7060, 7041, 7241, 7740, 7470/7471

A-,S- Methods from "Sampling and Analysis for Hazardous Waste Combustion", February 1984.
M - Methods from "Methods for Chemical Analysis of Water and Wastes", March 1979.
SW - Methods from "Test Methods for Evaluating Solid Waste", Third Edition, November 1986.

TABLE 4-3. SAMPLING AND ANALYSIS SUMMARY FOR ROSE TOWNSHIP-DEMODE ROAD CONTINUOUS RUN

Source	Sample Collection Frequency	Sampling Method	Analysis Parameters	Analysis Method
Solid Waste Feed	Grab sample every 30 minutes and composite	Scoop or Trowel (S007)	PCB	EPA 680
			PCDD/PCDF	SW 8280
			Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
			TCLP	SW 8270, 8240, 6010, 7060, 7041, 7421
			EP Toxicity	SW 1310
			Moisture, Ash	A001
			Ultimate	A003
			Density	GRAV/VOL
Furnace Ash	Sample at end of test period	Trier (S006)	PCB	EPA 680
			PCDD/PCDF	SW 8280
			Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
			EP Toxicity	SW 1310
			TCLP	SW 8270, 7420, 6010, 7060, 7041, 7421
			Moisture, Ash	A001
			Chlorine	A003
			Density	GRAV/VOL
Scrubber Water	Sample at end of test period composite	Dipper (S002)	PCB	EPA 680
			PCDD/PCDF	SW 8280
			pH	SW 9040
			Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
			EP Toxicity	SW 1310
			TCLP	SW 8270, 7420, 6010, 7060, 7041, 7421
			Total Organic Carbon	M415.1
			Total Suspended Solids	M160.1
			Total Dissolved Solids	M160.2
Scrubber Solids	Sample at end of test period composite	Dipper (S002)	PCB	EPA 680
			PCDD/PCDF	SW 8280
			Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
			EP Toxicity	SW 1310
			TCLP	SW 8270, 8240, 6010, 7060, 7041, 7421
			Ash	A003
			Ultimate	A003
			Density	GRAV/VOL

(Continued)

TABLE 4-3. (Continued)

Source	Sample Collection Frequency	Sampling Method	Analysis Parameters	Analysis Method
Ambient Air	Continuous over 12-hour period; one upwind and one downwind	PUF	PCB	EPA 680
	Continuous over 12-hour period; one upwind and one downwind	Hi-Volume sampler	Metals	SW 6010, 7060, 7041, 7421, 7740, 7470/7471
Stack Gas and Primary Furnace Offgas	Continuous	Continuous emission monitors	O_2 CO CO_2 THC NOx	Paramagnetic NDIR NDIR FID Chemiluminescence
	Composite of 1-3 hour period	EPA Methods	Particulate Matter Volumetric Flowrate Moisture	EPA Method 5 EPA Methods 1-4 EPA Method 4

A-,S- Methods from "Sampling and Analysis for Hazardous Waste Combustion", February 1984.
M - Methods from "Methods for Chemical Analysis of Water and Wastes", March 1979.
SW - Methods from "Test Methods for Evaluating Solid Waste", Third Edition, November 1986.

4.3 Sampling Locations

This subsection presents a brief description of each location where samples will be collected. The sampling locations are depicted in Figure 4-1.

The streams which will be sampled are:

1. Stack Gases;
2. Primary Furnace Offgas;
3. Solid Waste Feed;
4. Furnace Ash;
5. Scrubber Water;
6. Scrubber Solids;
7. Scrubber Make-Up; and
8. Ambient Air.

The Shirco Portable Pilot Test Unit demonstration tests at the Rose Township site will run for 10 days, 12 hours per day.

4.3.1 Stack Gases

Gases exiting the scrubber unit are propelled by an induced draft fan and exhausted through a 4.0-inch ID stack. The stack extends through the roof of the trailer to an elevation of 23.6 feet above grade (10 feet above the trailer roof). The stack is equipped with sampling ports accessible from the trailer roof. Because of the size of the exhaust stack, the duct will not be traversed. All samples will be collected at a single point in the center of the pipe.

About 3 feet above the roof of the trailer is a 3-inch flanged port and a 1-inch sample tap. Approximately 3 feet above these ports is another 3-inch flanged port. The two 3-inch ports above the trailer roof will be used to collect SASS, Method 5, and Modified Method 5 samples; see Figure 4-2.

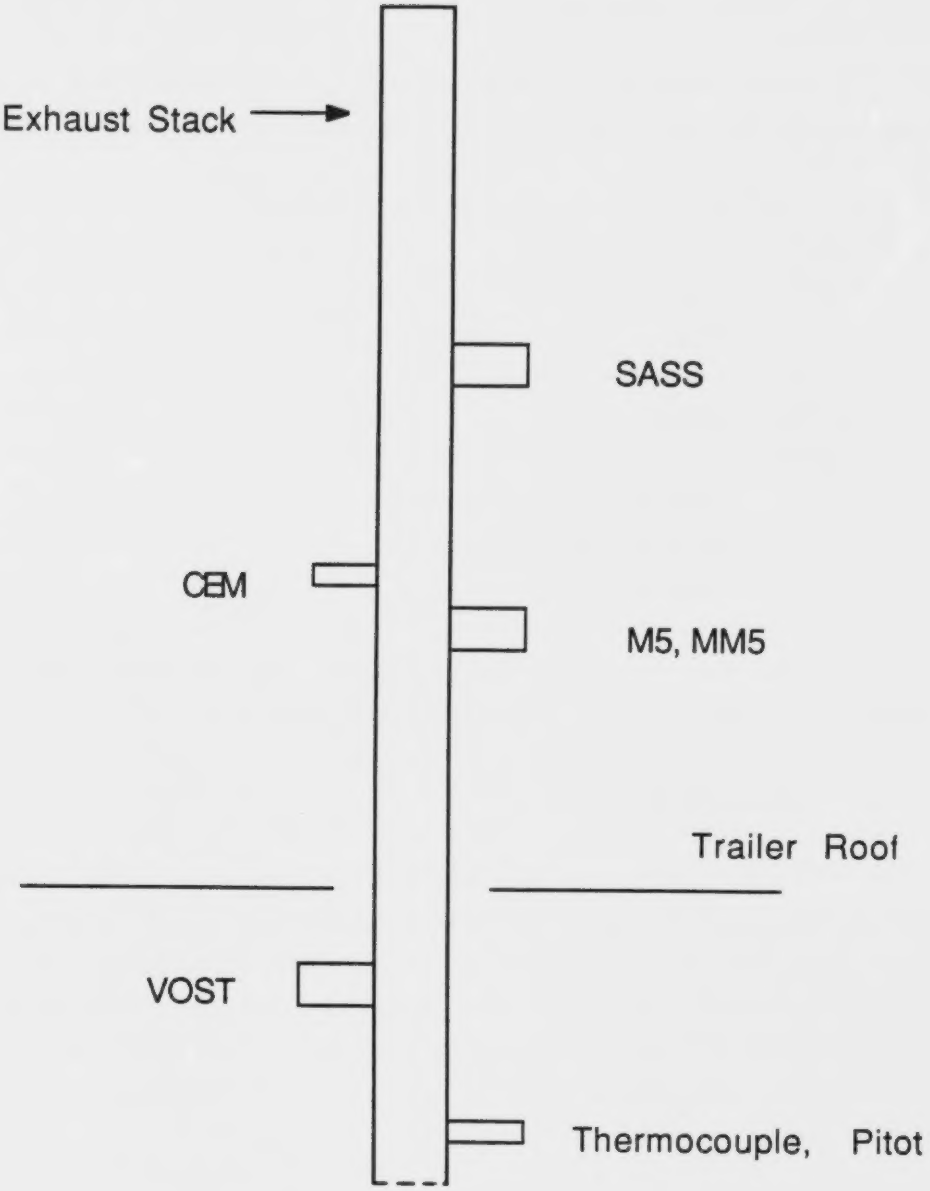

Exhaust Stack
SASS
CEM
M5, MM5
Trailer Roof
VOST
Thermocouple, Pitot

Figure 4-2. Schematic of Exhaust Stack Showing Location of Sampling Ports

Prior to the point at which the exhaust stack passes through the trailer roof is another 2-inch port accessed through a bulkhead in the trailer. VOST samples will be collected at this location.

All probe entries or unused ports will be sealed using packing glands or port covers during sampling episodes to minimize air in-leakage. The 1-inch port above the stack has been projected as the CEM sampling tap. However, due to the potential for in-leakage through the 3-inch port at the same location during sampling activities, it is recommended that a sampling tap (1-inch or 1/2-inch) be installed in the exhaust pipe upstream of the VOST sampling port.

Another consideration proposed is the installation of yet another tap (2-inch) to install a fixed pitot tube and thermocouple in the exhaust stack to continuously monitor stack flue gas velocity. Because of the small exhaust duct size, following conventional sampling protocol using a nozzle, pitot, and thermocouple at the sampling point in the duct would overly obstruct flow and potentially bias data. Under the proposed approach, flue gas velocity will be monitored upstream of the sampling locations in an unobstructed flow region. These data will be used to determine isokinetic sampling rates. SASS, Method 5, and Modified Method 5 probes will not include a pitot or thermocouple which will also accommodate application of a packing gland to seal the probe to the exhaust stack port.

4.3.2 Primary Furnace Offgas

The offgas from the primary furnace passes through a 2-feet x 2-feet duct into the secondary combustion chamber. There are two 4-inch 150 lb flanged ports available for sampling access located on either side of the duct. An additional 1-inch port is also available for collection of sample gas for the continuous monitors. Sampling access is via 1-foot x 2-feet bulkheads on either side of the trailer and in line with the ports. The duct walls are about 3-feet from the exterior wall of the trailer.

4.3.3 Solid Waste Feed

The waste feed consists of contaminated soils. Grab samples will be collected by Shirco personnel every 30 minutes during the tests from the incinerator feed bucket using a scoop. Two liters of sample will be collected during the SITE test burn and during the continuous run tests. Samples for analysis will be composited in 1 liter amber glass jars with a teflon lined cap.

4.3.4 Furnace Ash

Ash samples will be collected from the ash collection drum at the end of each test period. The samples will be collected and composited in 1 liter amber glass jars with teflon-lined lids.

4.3.5 Scrubber Water

The scrubber water recirculates through a 50 gallon drum outside of the incinerator trailer. Samples of the scrubber water will be taken at the end of each test period from the drum using a dipper. Six liters of scrubber water will be collected in amber glass jars with teflon-lined lids, along with three 40 mL VOA vials.

4.3.6 Scrubber Solids

Particulate matter and salts are removed from the exhaust gas by a venturi scrubber. These solids settle out of the scrubber water and collect on the floor of a 50 gallon recirculation drum. Scrubber solids will be collected at the end of each test period using a dipper. Approximately 2,500 grams of sample are necessary to achieve all analytical requirements.

Based upon discussions with process operating personnel, it is likely that inadequate solids will be collected during a test period to accommodate analytical requirements. If adequate sample amount is unavailable, then analysis of scrubber solids will be eliminated. During each test, several scrubber water samples will be collected and analyzed for total suspended solids to evaluate the solids loading of the scrubber water.

4.3.7 Scrubber Make-Up Water

Samples of the scrubber make-up water will be collected from a tap in the line running from the holding tank to the scrubber intake port at the end of each test period. Six liters of scrubber make-up water will be collected in amber glass jars with teflon-lined lids, along with three 40 mL VOA vials.

4.3.8 Ambient Air

Ambient PCB and metals concentrations will be measured upwind and downwind of the incineration area during the demonstration tests. Samples will be collected during the SITE testing and during the six days of the continuous run testing.

4.3.9 Process Data

Shirco Portable Pilot Test Unit operating personnel will record process data during the test periods at hourly intervals. Figure 4-3 presents the process data sheet which will be used to record the data.

4.4 Sampling Procedures

This section describes in detail the sampling methods and procedures to be used by Radian during the Shirco incinerator demonstration test for

DATE ________ OPERATOR ______________

	TAG NO.	UNITS	TIME				
TEMPERATURE DATA							
PCC 1							
PCC EXHAUST							
SCC EXHAUST							
SCC MIDDLE							
SCRUBBER QUENCH #							
ASH DISCHARGE							
EXHAUST STACK							
FLOW DATA							
SOLID WASTE FEED RATE							
QUENCH WATER							
QUENCH RECYCLE							

Figure 4-3. Recommended Process Data Recording Sheet

DATE ________ OPERATOR ______________

	TAG NO.	UNITS	TIME				
VENTURI RECYCLE							
ASH QUENCH RATE							
PCC AIR FLOW							
SCC AIR FLOW							
SCC FUEL FLOW							
VENTURI PRESSURE DATA							
VENTURI ΔP							
PCC PRESSURE							
SCC PRESSURE							
ID FAN SUCTION							
MISCELLANEOUS DATA							
STACK GAS VELOCITY							
SCRUBBER SOLUTION pH							
SCC O_2							
STACK CO2							
STACK CO							
BELT SPEED							
ELECTRIC POWER TOTAL							
ELEC. PWR.-PCC/HEPC							

Figure 4-3. (Continued)

collection of samples of the stack gas, liquid and solid samples and sampling of ambient air. Process data will also be collected by Shirco personnel.

4.4.1 Stack Gas and Primary Furnace Offgas Sampling Procedures

Sampling procedures for the stack gas and primary furnace offgas are described in the following sections.

4.4.1.1 EPA Method 5

The stack gas and primary furnace offgas will be sampled for measurement of particulate matter, HCl, volumetric flow rate, moisture, and metals using an EPA Method 5 sampling train. The method will be modified by replacing water with 0.1 N NaOH in the impingers to collect HCl. The Method 5 sampling train is shown in Figure 4-4.

On the stack, the method will be further modified by locating the pitot tube and thermocouple for gas velocity measurement at a location in the stack separate from the extraction points. This approach is necessitated by the small size of the stack diameter and obstruction of gas flow if a nozzle, pitot, and thermocouple are colocated. The flue gas flow monitoring technique is in accordance with EPA Method 2A.

Based on the Method 5 technique, a sample of particulate-laden flue gas is withdrawn isokinetically using a gooseneck nozzle and heat-traced, glass-lined probe. The particulate matter is collected on a glass fiber filter maintained at a temperature in the range of 248 ±25°F. The particulate mass is determined gravimetrically from the residues collected on the filter and in the probe and associated glassware prior to the filter. After the filter, the flue gas enters a chilled impinger train where HCl is collected in the first two Greenburg-Smith impingers containing 200 mL of 0.1 N NaOH. A third dry impinger follows to collect condensate or mist carryover from the previous impingers. The third impinger will be a modified Greenburg-Smith

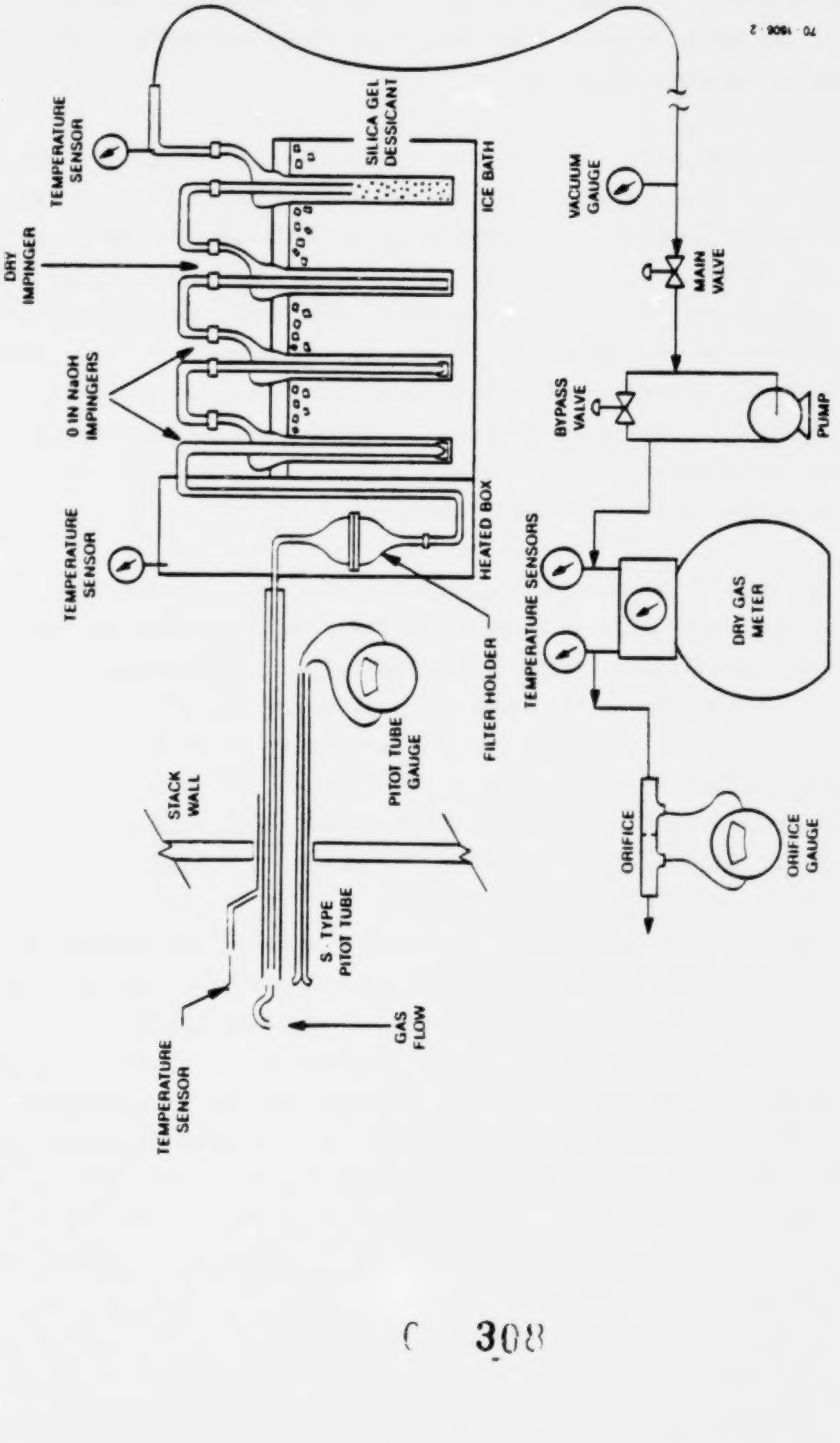

TEMPERATURE SENSOR
DRY IMPINGER
0.1N NaOH IMPINGERS
TEMPERATURE SENSOR
SILICA GEL DESSICANT
ICE BATH
HEATED BOX
FILTER HOLDER
PITOT TUBE GAUGE
STACK WALL
S-TYPE PITOT TUBE
GAS FLOW
TEMPERATURE SENSOR
70-1606-2
VACUUM GAUGE
MAIN VALVE
BYPASS VALVE
PUMP
TEMPERATURE SENSORS
DRY GAS METER
ORIFICE
ORIFICE GAUGE

Figure 4-4. Schematic of the EPA Method 5 Sampling Train for Particulate Matter and HCl

type. The fourth impinger will contain a known weight of silica gel desiccant to collect remaining moisture. A pump and dry gas meter are used to control and monitor the gas flow rate. The data sheet shown in Figure 4-5 will be used to record sampling data.

During collection of EPA Method 5 samples, S-type pitot measurements are taken in the flue gas duct to determine the isokinetic sampling rate. The pitot differential pressure measurements, along with the flue gas composition (CO_2, O_2, N_2, H_2O) and cross-sectional area of the duct, are also used to determine the volumetric flue gas flow rate. Note that stack gas velocity will not be measured at the nozzle. During the sampling runs, grab samples of the stack gas will be collected to determine the concentrations of CO_2 and O_2 directly and N_2 by difference, in accordance with EPA Reference Method 3 protocol. The moisture content of the sample gas will be measured during the runs following EPA Reference Method 4 protocol.

At the end of the sampling period, the nozzle, probe liner, and glassware preceding the filter housing are rinsed with acetone and deionized water to remove particulate matter. The resulting wash is evaporated and the mass of particulate residue is determined gravimetrically. The glass fiber filter is removed from the filter holder, desiccated for 24 hours, and weighed to determine the mass of particulate on the filter. The total mass of particulate present on the filter and in the probe wash is then divided by the total volume of gas sampled to determine the particulate loading.

The impingers used during particulate sampling are weighed before and after sampling to determine the moisture content of the flue gas. The HCl concentration of the flue gas is determined by analyzing two sodium hydroxide impingers for chloride. Since the impinger solutions are caustic, CO_2 will also be removed. To account for the CO_2 removal, the 0.1 N NaOH impinger solutions will also be analyzed for carbonate. The metered gas sample volume will be adjusted using the carbonate analytical values.

A01 (1-3) Plant Name>____

Sampling Location>____________________ Sample Ident.>________________

A02 (1-3) Date __________(MMDDYY) Time Start__________(HHMM) Time Finish__________(HHMM)

Duct Dimensions________x________ft. or Diameter ____________________ft.

PTCF__________ DGMCF__________ Test Duration____________________min.

A03 (1-3) Nozzle Dia.______________inches % CO______________ % N_2 ______________

Bar Press.______________"Hg % CO_2______________ % H_2 ______________

Static Press.______________"H_2O ____ % O_2______________ % CH_4 ______________

A04 (1-3) Initial leak rate__________cfm Condensed Water____________________gms

Final leak rate__________cfm Filter wt. gain____________________gms

Operator initials__________ Probe wash wt. gain____________________gms

	Pt.	Clock Time	Dry Gas Meter Reading ft³	ΔP in. H_2O	ΔH in. H_2O	Stack Temp. °F	Dry Gas Meter Temperature		Hot Box Temp. °F	Last Impinger Temp. °F	L. Vac. in. Hg Gauge
							Inlet °F	Outlet °F			
A05											
A06											
A07											
A08											
A09											
A10											
A11											
A12											
A13											
A14											
A15											
A16											
A17											
A18											
A19											
A20											
A21											
A22											

Console______________ Filter__________________ Initial Wt.______________gms

Sample Analysis____

Weather____

____

A23 Remarks____

____

Figure 4-5. Source Sampling Data Sheet

The particulate matter collected on the glass fiber filter will be analyzed for metals. The measured metals concentration along with the particulate loading and flue gas flow rate will be used to determine the emission rates of those metals.

4.4.1.2 Source Assessment Sampling System

The Source Assessment Sampling System (SASS) will be utilized during this program for the determination of semivolatile priority pollutants (plus 10 highest peaks) [SV-PP+10], PCDD/PCDF, and PCBs. XAD-2 will be the adsorbent resin in the train. Samples for PCBs will be collected in a separate SASS train from the samples collected for SV-PP+10 and PCDD/PCDF analysis.

The SASS protocol is outlined in "Modified Method 5 Train and Source Assessment Sampling System Operator's Manual," February 1985, by the U.S. Department of Commerce (NTIS) and "Test Methods for Evaluating Solid Waste", Method 0020, SW-846, Third Edition, November 1986. The SASS train is shown in Figure 4-6.

The sampling system consists of a heated probe, a heated filter, a condenser, a sorbent module, and a pumping and metering unit. Because of the low particulate loading in the gas stream, the three cyclones of the SASS have been removed from the train for sampling. The probe is a stainless steel sheath which contains a heat-traced stainless steel sample liner. A gooseneck nozzle of proper size to allow near-isokinetic sample collection is attached to the probe. The flue gas velocity is measured by an S-type pitot, using either an oil manometer or Magnehelic® differential pressure gauge to measure the ΔP of the pitot. The probe is connected directly to a heated enclosure which houses a high efficiency glass fiber filter. The enclosure is maintained at a temperature of 400°F.

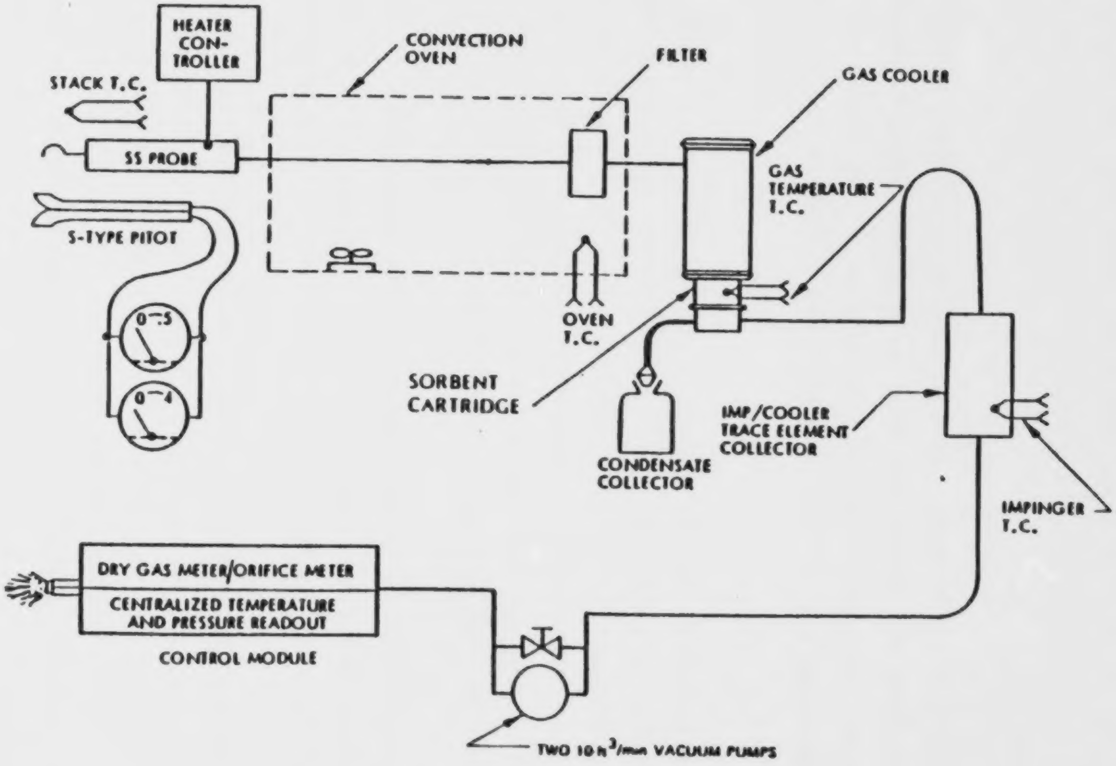

HEATER
CON-
TROLLER
STACK T.C.
SS PROBE
S-TYPE PITOT
0–.5
0–4
CONVECTION
OVEN
FILTER
OVEN
T.C.
SORBENT
CARTRIDGE
GAS COOLER
GAS
TEMPERATURE
T.C.
CONDENSATE
COLLECTOR
IMP/COOLER
TRACE ELEMENT
COLLECTOR
IMPINGER
T.C.
DRY GAS METER/ORIFICE METER
CENTRALIZED TEMPERATURE
AND PRESSURE READOUT
CONTROL MODULE
TWO 10 ft³/min VACUUM PUMPS

Figure 4-6. Source Assessment Sampling System Train

From the heated filter, the sample gas enters a water-cooled condenser, followed by the XAD-2 sorbent module for collection of vapor phase organic material. A trap follows the sorbent module which will collect the aqueous condensate.

After the condensate trap are three dry impingers to collect any mist carryover from the condensate trap and a final impinger containing a desiccant to dry the sample gas prior to metering. The sample gas is drawn by two double diaphragm pumps, and the sample gas volume is measured using a dry gas meter.

The design of the SASS train precludes traversing of the stack. Sample collection is performed at a single point of average gas velocity based on previously-determined velocity traverse data. The SASS probe includes an S-type pitot and thermocouple to measure sample gas velocity to determine the isokinetic flowrate. As with the Method 5 train, the probe pitot and thermocouple will not be used to sample the stack gas.

Conventionally, 30 cubic meters (1,060 cubic feet) of gas are sampled. Sample collection time will be approximately three to six hours. The allowable isokinetic sampling rate for the SASS is -70 to ±150 percent.

Sampling train preparation and sample retrieval will be performed indoors in a controlled environment to reduce the possibility of sample contamination. Prior to assembly, each component of the sampling train will be thoroughly rinsed with methylene chloride. The glass fiber filter will be pre-extracted with methylene chloride. All connections in the sampling train will be installed dry, or where possible, with Teflon to reduce the possibility of contamination. Once the sampling train has been assembled, the nozzle and exit port will be sealed with methylene chloride-rinsed foil. The data sheet presented in Figure 4-7 will be used to document pertinent source sampling information during the collection of SASS samples.

A01 (1-3) Plant Name>____

Sampling Location>____________________ Sample Ident.>____________

A02 (1-3) Date __________(MMDDYY) Time Start__________(HHMM) Time Finish__________(HHMM)

Duct Dimensions________x________ft. or Diameter ________________ft.

PTCF__________ DGMCF__________ Test Duration________________min.

A03 (1-3) Nozzle Dia.__________inches % CO__________ % N_2 __________

Bar Press.__________"Hg % CO_2__________ % H_2 __________

Static Press.__________"H_2O ____ % O_2__________ % CH_4 __________

A04 (1-3) Initial leak rate__________cfm Condensed Water________________gms

Final leak rate__________cfm Filter wt. gain________________gms

Operator initials__________ Probe wash wt. gain________________gms

	Pt.	Clock Time	Dry Gas Meter Reading ft³	ΔP in. H_2O	ΔH in. H_2O	Stack Temp. °F	Dry Gas Meter Temperature		Hot Box Temp. °F	Last Impinger Temp. °F	L. Vac. In. Hg Gauge	Condenser Outlet Temp.
							Inlet °F	Outlet °F				
A05												
A06												
A07												
A08												
A09												
A10												
A11												
A12												
A13												
A14												
A15												
A16												
A17												
A18												
A19												
A20												
A21												
A22												

Console__________ Filter__________ Initial Wt.__________gms

Sample Analysis______________________________

Weather______________________________

A23 Remarks______________________________

Figure 4-7. SASS Sampling Data Sheet

After sample collection, the ends of the sampling train will once again be sealed with solvent-rinsed foil and returned to the clean-up area for sample retrieval. The filter will be recovered and placed in a methylene chloride-rinsed glass petri dish. Aqueous condensate collected in the condensate trap and in the impingers will be transferred to methylene chloride-rinsed amber glass bottles with Teflon®-lined closures. All components of the sampling train, from the nozzle through the sorbent module and including the probe, filter glassware, and impinger glassware, will be thoroughly rinsed and quantitatively recovered.

4.4.1.3 Soluble Chromium

Soluble chromium (hexavalent chromium) sampling will be conducted according to the procedures (with modifications) currently being used by the EPA's Emission Measurement Branch (EMB) for sampling hexavalent chromium emissions from municipal waste incinerators. This procedure involves the use of an EPA Method 5 sampling train with the following modifications:

- 0.1 N NaOH impingers in place of water;
- No filter;
- A glass nozzle in place of stainless steel;
- 0.1 N NaOH rinse to recover the sample; and
- Minimal 0.1 N NaOH in the sample recovery process.

The nozzle, probe liner, and pre-impinger glassware rinse will be added to the impinger catches and the sample analyzed by atomic absorption spectroscopy. The soluble chromium sampling train will be run approximately 4 hours at a point of average flue gas velocity to achieve adequate analytical sensitivity.

4.4.1.4 VOST

The stack gas and primary furnace offgas will be sampled for volatile organic compounds and priority pollutants (plus the 10 highest peaks) using the VOST (Volatile Organics Sampling Train). The VOST protocol (Method 0030) is outlined in "Test Methods for Evaluating Solid Waste", SW-846, Third Edition, November 1986. The VOST is designed to collect volatile organics with boiling points less than approximately 100°C using a pair of adsorbent resin traps in series.

A schematic diagram of the VOST sampling train is shown in Figure 4-8. Volatile organics are removed from the gas in sorbent resin traps containing Tenax and Tenax-charcoal maintained at 20°C. The first resin trap contains Tenax and the second trap contains Tenax followed by petroleum-based charcoal. After sampling, the resin traps are sealed and returned to the laboratory for analysis. A 20-L sample of gaseous effluent is collected at a flow rate of 1.0 L/min using a glass-lined probe. A dry gas meter is used to measure the volume of gas passed through the pair of traps. An example VOST source sampling data sheet is shown in Figure 4-9.

During the test, a VOST run will consist of collecting six pairs of traps, with each pair of traps exposed to sample gas for 20 minutes at the 1.0 L/min flow rate. After daily sampling, two 40 mL VOA vials will be used to collect the aqueous condensate collected in the condenser. Three analyses will be performed on the six resin trap pairs and on one of the aqueous condensate vials. The samples will be collected at a fixed point of average gas velocity in the duct. Isokinetic sampling is not required since all volatile POHCs are in the gas phase.

Descriptions of the train components are given below:

Probe--The probe is lined with borosilicate or quartz glass. The glass liner provides an inert surface so that the sample gas does not undergo changes in composition. The liner is equipped with a

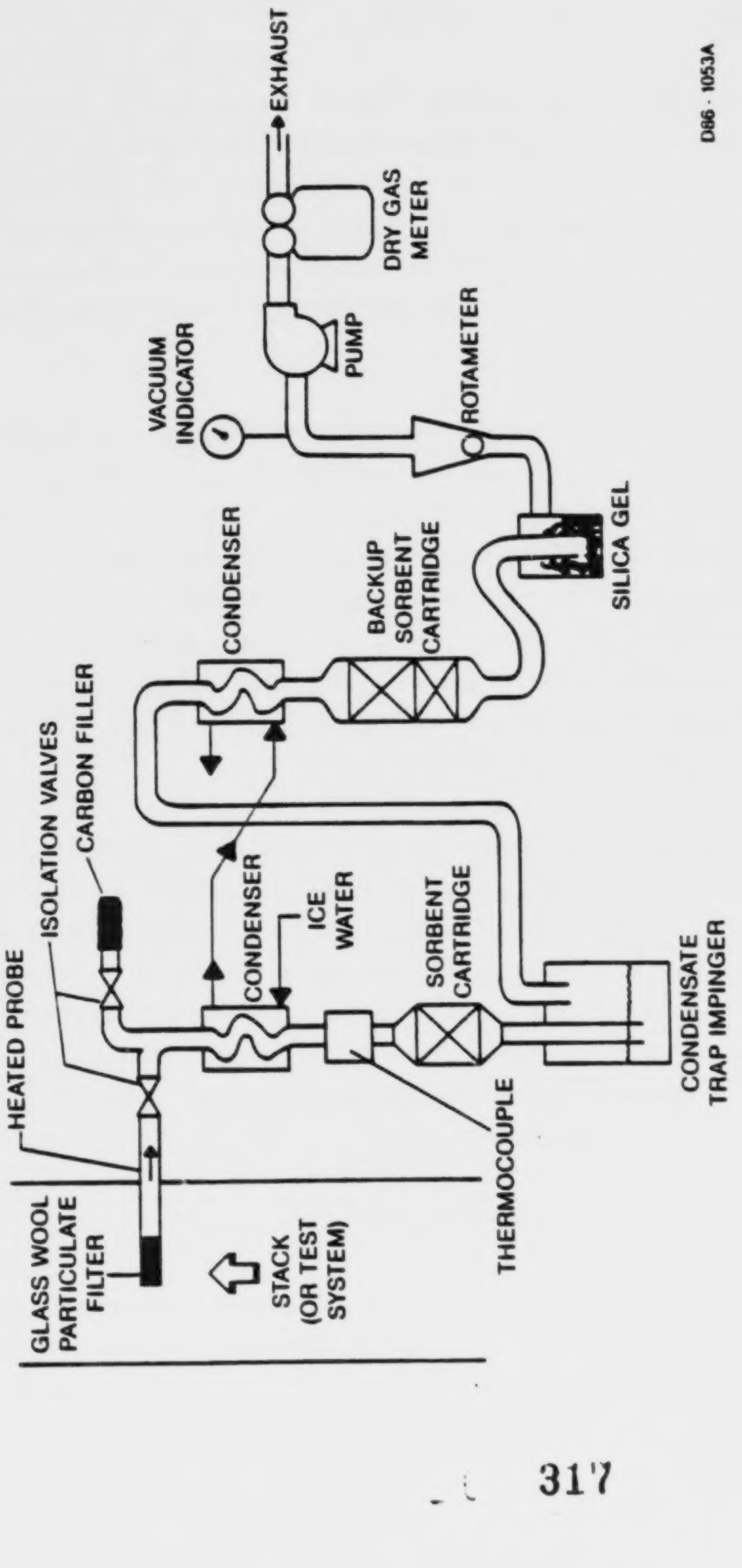

GLASS WOOL PARTICULATE FILTER
STACK (OR TEST SYSTEM)
HEATED PROBE
ISOLATION VALVES
CARBON FILLER
THERMOCOUPLE
CONDENSER
ICE WATER
SORBENT CARTRIDGE
CONDENSATE TRAP IMPINGER
CONDENSER
BACKUP SORBENT CARTRIDGE
SILICA GEL
VACUUM INDICATOR
ROTAMETER
PUMP
DRY GAS METER
EXHAUST
D86-1053A

Figure 4-8. Schematic Diagram of VOST Sampling Train

VOST Field Data Sheet

Plant ____________________
Date ____________________
Location ____________________
Console # ____________________
DGMCF ____________________

Field Blank ID: Tenax ____________________
Tenax/Charcoal ____________________
Operator ____________________
Barometric Pressure ____________________
Sampling Duration ____________________

Pair No.	Leak Check	Rotameter (l/min)	DGM Reading (L)		Time	Probe Temp.	Condenser Outlet Temp.	Meter Temp.		Meter Pressure	Pump Vacuum (in - Hg)
			Initial	Final				Inlet	Outlet		

Condensate Samples:
Run No. ____________________
Sample ID ____________________

Comments:

Figure 4-9. VOST Source Sampling Data Sheet

connecting fitting that is capable of forming a leak-free connection without sealing greases. The probe is maintained at a temperature of at least 130°C prior to the first condenser.

Isolation Valve--The isolation valve is a greaseless stopcock with a glass bore and a sliding Teflon® plug with Teflon® wipers.

Condensers--The condensers are of sufficient capacity to cool the gas stream to 20°C or below prior to the first sorbent trap. The condensers are fitted with leak-free connections.

Sorbent Traps--The sorbent traps are of the inside-inside type as illustrated in Figure 4-10. The cartridge is approximately 1.6 centimeters I.D. and 10 centimeters long. The first trap in the series is packed with approximately 1.6 grams of Tenax GC resin. The second trap is packed with approximately 1 gram each of Tenax GC resin and petroleum-based charcoal. The second trap is packed so that the sample gas passes through the Tenax layer first and then through the charcoal layer.

Metering System--The metering system consists of vacuum gauges, a leak-free pump, a calibrated rotometer, a dry gas meter accurate to within ±2 percent at the required sampling rate, and related valves and equipment. A thermocouple is placed on the exterior glass surface at the exit of the first condenser to monitor and maintain the gas temperature entering the first sorbent trap at 20°C or less.

The glass resin tubes and condensers will be cleaned with a nonionic detergent in an ultrasonic bath, rinsed in HPLC-grade distilled water, and dried at 110°C. Resin tubes will be capped with end caps and stored in cleaned glass containers with cotton cushioning. Condensers and other glassware will also be capped with appropriate end caps prior to use.

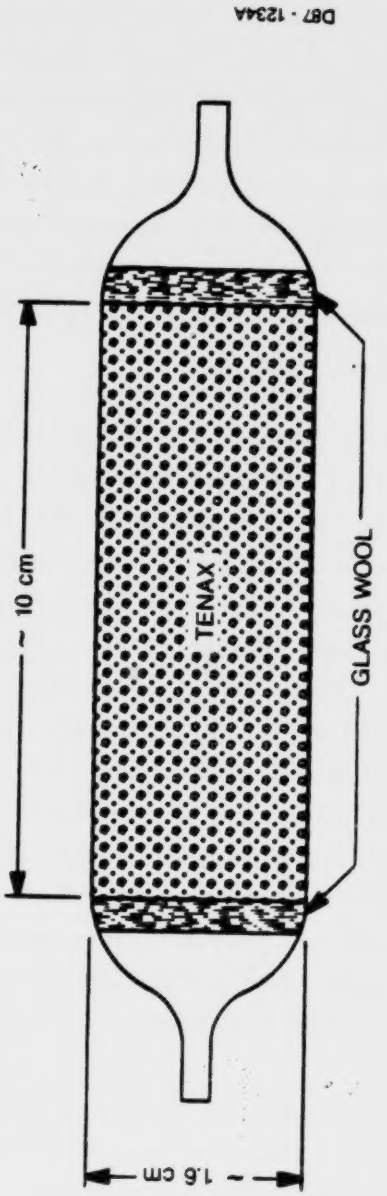

D87 - 1234A
TENAX
GLASS WOOL
~ 10 cm
~ 1.6 cm

Figure 4-10. Inside-Inside VOST Cartridge

At Radian laboratories before the sample tubes are sent to the field, the Tenax and Tenax-charcoal cartridges will be loaded and leak-checked using the following procedures. The traps are marked to show the flow direction for sampling, so that when the Tenax-only traps are returned to the laboratory for analysis, they can be spiked for QA/QC purposes and connected to the analytical system in the proper orientation. The Tenax-only traps contain 1.6 grams of Tenax, and the Tenax-charcoal traps contain 1 gram of Tenax and 1 gram of charcoal. This appears visually as 2/3 Tenax and 1/3 charcoal by volume. The traps are conditioned prior to use according to Method 0030 protocol.

The virgin Tenax is prepared by Soxhlet extraction with methanol and then pentane for 24 hours each and dried in a vacuum for 6 hours at 50°C. (The charcoal is prepared in the same manner.) The tubes are then packed individually and thermally conditioned for 24 hours at 190°C with organic-free nitrogen (or helium) flowing at 30 mL/min. The effluent from the conditioning system is monitored with a flame ionization detector to check for emissions of volatile organic compounds. Conditioning is repeated until no emissions are observed at the detection limit of 2 ng of benzene and/or toluene by cyro-focusing GC/FID detector. One out of every 10 tubes is screened further by treating it as a sample and performing an analytical thermal desorption step. Subsequent conditioning (after sampling and analytical desorption) will include only the thermal steps unless the tubes have experienced unusually high levels of organic compounds, in which case, both Soxhlet extraction and thermal conditioning are measured with the tube having the lowest flow being selected for the additional screening step.

After sample collection, the cartridges will be leak-checked again. After leak checks have been completed at the end of sampling with each pair of traps, the traps will be sealed with their end caps and returned to their respective glass containers for storage and transport. During transportation and storage, the traps will be maintained at 4°C until analysis.

4.4.1.6 Molecular Weight

Stack gas and primary furnace offgas will be collected in tedlar bags for determination of O_2 and CO_2 concentrations. The sample will be extracted through a stainless steel probe and passed through a silica gel impinger to dry the gas before collection in the tedlar bag. Analysis will be conducted by EPA Method 3 (Orsat).

4.4.1.7 Continuous Emission Monitors (CEMs)

CEMs will be used during the demonstration test to monitor the concentrations of CO, CO_2, O_2, NO_x, and THC in the stack gas and primary furnace offgas using one set of instruments. The instruments will be switched between the two sources every 30 minutes.

A stainless steel probe will be inserted at the stack gas and primary furnace offgas sampling locations. Gas will be withdrawn and transported via separate heat-traced (at 300°C) Teflon® sample lines to the instrumentation area. There the stack gas and primary furnace offgas sample lines will be fed into a three-way valve. The selected stream will be conditioned prior to analysis to remove both particulate matter and water.

Gas will first enter an impinger train having a series of short-stemmed impingers (as condensers) immersed in an ice bath. After the impinger train, particulate matter and mist will be removed by a glass fiber filter. After filtration, the gas will be further dried using a Perma-Pure dryer which utilizes a water vapor permeable membrane.

The gas will be drawn by a Teflon®-coated diaphragm pump located between the filter and the Perma-Pure dryer. Flue gas for the five instruments will be drawn from a manifold downstream of the pump.

A brief description of each CEM instrument is provided below.

Carbon Monoxide

A Bendix Model 85-105CA analyzer will be used to measure CO concentration in the stack gas and primary furnace offgas. This instrument is a non-dispersive infrared (NDIR) analyzer which measures the concentration of CO by infrared absorption at a characteristic wavelength.

Carbon Dioxide

To measure the CO_2 concentration in the stack and primary furnace gas, an MSA Model 303 non-dispersive infrared (NDIR) analyzer will be used. This instrument measures the concentration of CO_2 by infrared absorption at a characteristic wavelength.

Oxygen

A Taylor Model 540A oxygen analyzer will be used to determine the O_2 concentration of the stack gas and primary furnace offgas. The Taylor 540A measures oxygen concentrations on the basis of the strong paramagnetic properties of O_2 compared to other compounds present in combustion gases. In the presence of a magnetic field, O_2 molecules become temporary magnets. The Taylor 540A determines the sample gas O_2 concentration by detecting the displacement torque of the sample test body in the presence of a magnetic field.

Nitrogen Oxides (NO_x)

A TECO Model 10 analyzer will be used to measure the concentration of NO_x present in the stack gas and primary furnace offgas. This instrument determines NO_x concentrations by converting all nitrogen oxides present in the sample gas to nitric oxide and then reacting the nitric oxide with ozone. The reaction produces a chemiluminescence proportional to the NO_x concentration in the sample gas. The chemiluminescence is measured using a high-sensitivity photomultiplier. The instrument has the capability to measure NO, NO_x and NO_2 by difference.

Total Hydrocarbons (THC)

A Beckman Model 400A will be used to continuously measure the concentration of hydrocarbons present in the stack and primary furnace gas. The analyzer utilizes a hydrogen flame ionization detector. The sensor is a burner where a regulated flow of sample gas passes through a flame sustained by regulated flows of a fuel gas and air. Within the flame, the hydrocarbon components of the sample stream undergo a complex ionization that produces electrons and positive ions. Polarized electrodes collect these ions, causing current to flow through an electronic measuring circuit. The ionization current is proportional to the rate at which carbon atoms enter the burner and is, therefore, a measure of the concentration of hydrocarbons in the original sample.

4.4.2 Solid and Liquid Sampling Procedures

Sampling procedures which will be used to collect samples from solid and liquid streams are described in this section. A summary of the solids and liquids sample volumes is presented in Table 4-4.

4.4.2.1 Solid and Liquid Sample Container Preparation

Sample containers for the solid and liquid samples must be organic free and sealed prior to receipt in the field. All sample bottles used for solid and liquid samples will be amber glass with Teflon® cap liners, cleaned to EPA protocols, and quality control analyzed (300 series).

All sample bottles will be purchased new from I-Chem Research, Inc. in Hayward, California. Each sample bottle that will be used to store samples for organic analysis will be pre-cleaned using the following procedure:

TABLE 4-4. SUMMARY OF LABORATORY ANALYSIS, SAMPLE QUANTITY AND PRESERVATION

Stream	Laboratory	Analysis Parameter	Sample Quantity	Sample Preservation
Solid Waste	Radian (Austin)	PCB	50 g	4°C
		PCDD/PCDF	50 g	4°C
		SV-PP+10	50 g	4°C
		Metals	100 g	
		TCLP	500 g	
		EP Toxicity	150 g	
		Density	500 g	
		V-PP+10	50 g	4°C
	Huffman Labs	Chlorine	1000 g	
		Moisture		
		Ash		
		HHV		
		Ultimate		
Scrubber Make-Up Water	Radian (Austin)	PCB	1 L	4°C
		PCDD/PCDF	1 L	4°C
		SV-PP+10	1 L	4°C
		Metals	1 L	pH<2 (HNO_3)
	Radian (Austin)	V-PP+10	3-40 mL VOA vials	4°C, no headspace
	Radian (Austin)	pH	1 L	4°C
		Chloride		
		Total Dissolved Solids		
		Total Suspended Solids		
		Total Organic Carbon	500 mL	4°C pH<2 (H_2SO_4)

(Continued)

TABLE 4-4. (CONTINUED)

Stream	Laboratory	Analysis Parameter	Sample Quantity	Sample Preservation
Scrubber Water	Radian (Austin)	PCB	1 L	4°C
		PCDD/PCDF	1 L	4°C
		SV-PP+10	1 L	4°C
		Metals	1 L	pH<2 (HNO_3)
		TCLP	1 L	4°C
		EP Toxicity		
		VP-PP+10	3-40 mL VOA vials	4°C, no headspace
	Radian (Austin)	pH	1 L	4°C
		Chloride		
		Total Dissolved Solids		
		Total Suspended Solids		
		Total Organic Carbon	500 mL	4°C, pH<2 (H_2SO_4)
Scrubber Solids	Radian (Austin)	PCB	50 g	4°C
		PCDD/PCDF	50 g	4°C
		SV-PP+10	50 g	4°C
		Metals	100 g	4°C
		TCLP	550 g	4°C
		EP Toxicity	150 g	
		Density	500 g	
		V-PP+10	50 g	4°C
	Huffman Labs	Chlorine	1000 g	
		Ash		
		Ultimate		

(Continued)

TABLE 4-4. (CONTINUED)

Stream	Laboratory	Analysis Parameter	Sample Quantity	Sample Preservation
Furnace Ash	Radian (Austin)	PCB	50 g	4°C
		PCDD/PCDF	50 g	4°C
		SV-PP+10	50 g	4°C
		Metals	100 g	
		TCLP	500 g	4°C
		EP Toxicity	150 g	
		Density	500 g	
		V-PP+10	250 g	4°C
	Huffman Labs	Chlorine	1000 g	
		Moisture		
		Ash		
Stack Gas	Radian (Austin)	Particulate		
		HCl		
		Metals		
		PCB (SASS)		4°C
		PCDD/PCDF (SASS)		4°C
		SV-PP+10 (SASS)		4°C
		Soluble Chromium		
	Radian (PPK)	V-PP+10 (VOST)		4°C
Primary Furnace Offgas	Radian (Austin)	Particulate		
		HCl		
		Metals		
		PCB (SASS)		4°C
		PCDD/PCDF (SASS)		4°C
		SV-PP+10 (SASS)		4°C
		Soluble Chromium		
	Radian (PPK)	V-PP+10		4°C
Ambient Air	Radian (Austin)	PCB (PUF)		4°C
		Metals (Filter)		

- Cleaned initially with a phosphate-free soap;
- Rinsed three times with tap water;
- Rinsed three times with deionized water;
- Rinsed with nitric acid;
- Rinsed three times with deionized water;
- Rinsed with methylene chloride;
- Baked in an oven for 6 hours at 200°C; and
- Allowed to cool and then capped.

4.4.2.2 Solid Sampling Procedures

Samples of the solid waste feed, furnace ash, and scrubber solids will be collected using a scoop, trier or dipper, as specified in U.S. EPA Methods S007, S006, and S002 of "Sampling and Analysis Methods for Hazardous Waste Combustion," February 1984. Furnace ash and scrubber solids will be collected at the end of each test period. Approximately 2,500 g of sample are necessary for analysis.

A uniform amount of solid waste feed will be collected every 30 minutes and placed in a sealed container. For each test, at least 2,500 g will be collected, and the total sample will be used for subsequent analysis. Since highly volatile compounds are not a critical element in this study, the chance loss of a small portion of these by handling during sampling (the waste feed will be intimately exposed to air several times before sampling) does not warrant extraordinary methods for sampling and handling in this study.

4.4.2.3 Liquid Sampling Procedures

Scrubber water and scrubber make-up water samples will be collected respectively using the dipper and tap sampling procedure specified in U.S. EPA Method S002 and S004, "Sampling and Analysis Methods for Hazardous Waste Combustion," February 1984. Liquid samples will be collected at the end of the test period. The sample tap will be flushed each time (allowed to flow briefly) before scrubber make-up water is collected. This will ensure that any stagnant accumulation of solids, or other contaminants that may be present in the tap, do not affect the sample integrity or its representation of the liquid process stream. Scrubber water will be collected directly from the accumulation drum. In excess of 6 liters of sample will be collected.

At each sample collection, three 40 mL VOA (void of air) sample vials will be collected for analysis of V-PP+10. The vials will be filled completely and no air bubbles allowed to remain in the bottles. The VOA samples will be composited at the time of analysis by syringe accumulation from the selected subsamples to provide one sample per test period for analysis.

4.4.3 Ambient Air

Ambient air both upwind and downwind of the test site will be sampled and analyzed for PCBs and metals during the SITE and Continuous Run programs. The upwind and downwind samplers will be located using on-site, continuous meteorological data, i.e., wind direction. The wind direction will be checked at least once per hour, and, since samplers should not be moved after sampling has begun, if the average wind direction deviates by more than 90°C, ambient sampling will be terminated for that period.

4.4.3.1 PCBs

Ambient concentrations of PCBs will be determined upwind and downwind of the site during the test programs using the PUF (polyurethane foam) technique. Monitoring will be performed using General Metals Works Inc. (GMW) Model PS-1 air samplers. Figures 4-11, 4-12, and 4-13 show the PUF sampler and its components.

The sampling system is housed in a standard aluminum high volume shelter and is designed to hold a 100 mm diameter quartz fiber filter at its

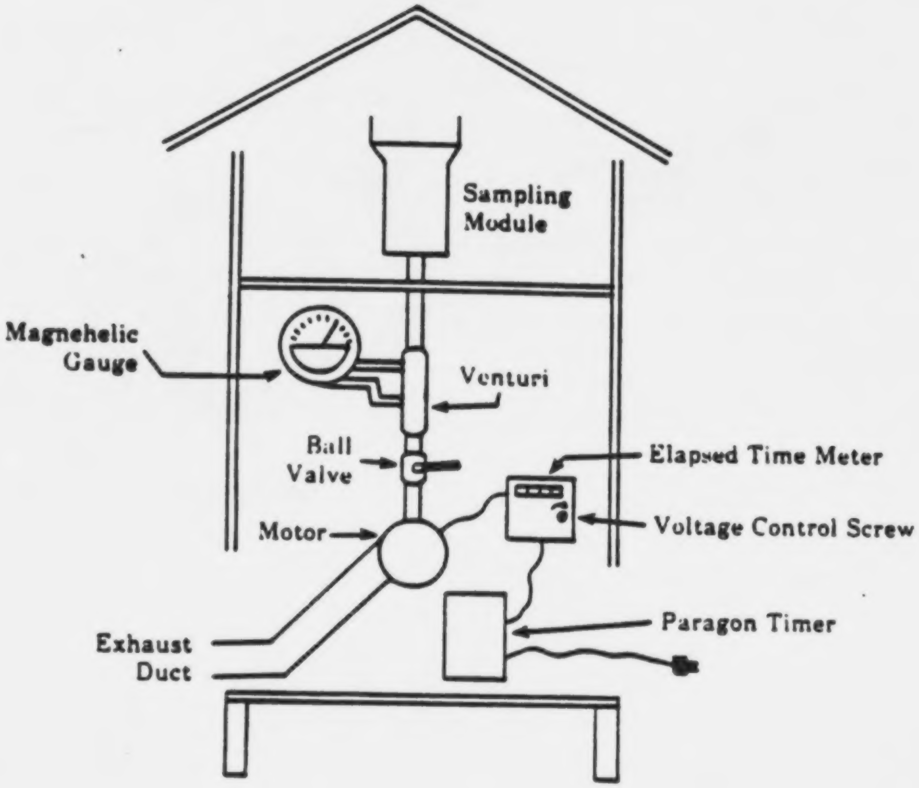

Sampling Module
Magnehelic Gauge
Venturi
Ball Valve
Elapsed Time Meter
Voltage Control Screw
Motor
Paragon Timer
Exhaust Duct

Figure 4-11. GMW PUF Sampler

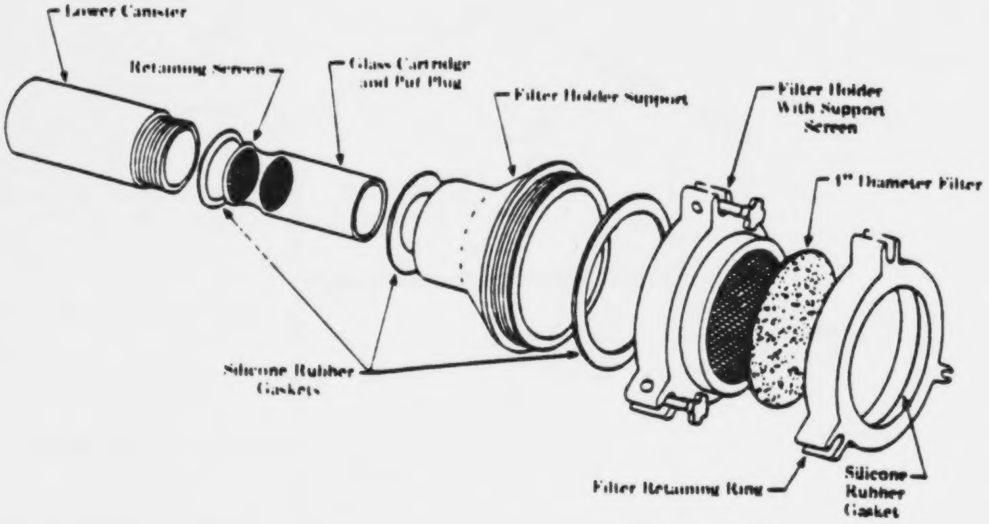

Lower Canister
Retaining Screen
Glass Cartridge and Puf Plug
Filter Holder Support
Filter Holder With Support Screen
4" Diameter Filter
Silicone Rubber Gaskets
Filter Retaining Ring
Silicone Rubber Gasket

Figure 4-12. PUF Sampler Components

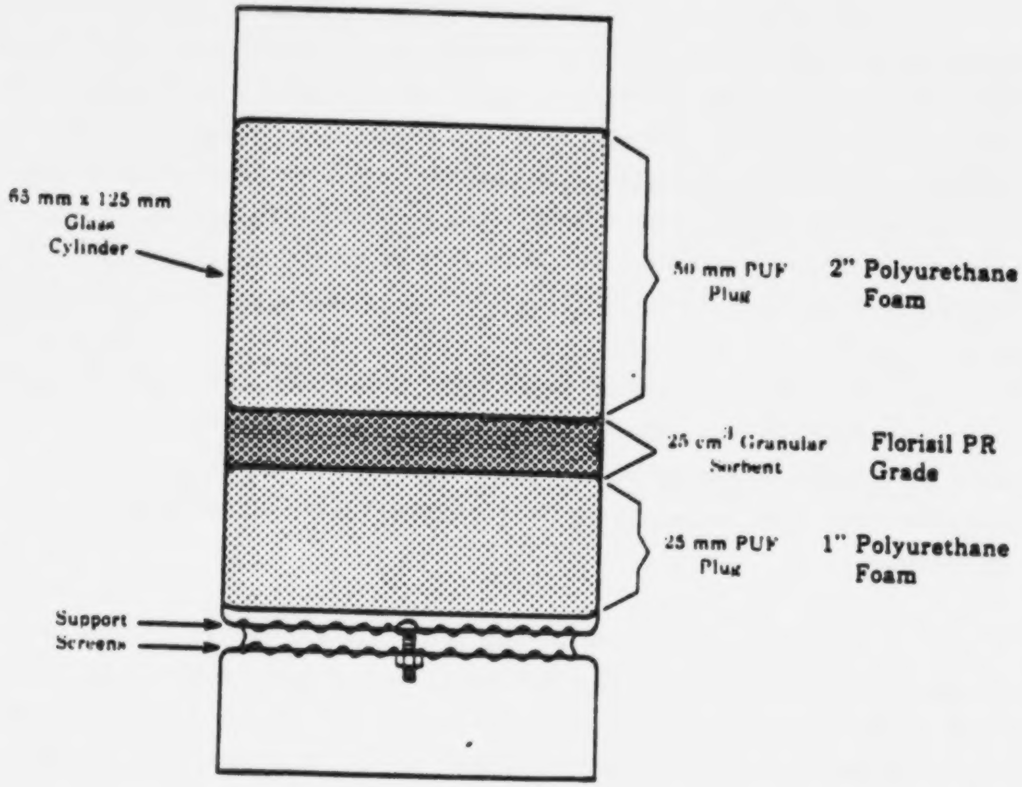

63 mm x 125 mm
Glass
Cylinder
50 mm PUF
Plug
2" Polyurethane
Foam
25 cm^3 Granular
Sorbent
Florisil PR
Grade
25 mm PUF
Plug
1" Polyurethane
Foam
Support
Screens

Figure 4-13. PUF Sampling Cartridge

inlet and a glass sampling cartridge in its lower canister. The glass cartridge is loaded with 25 mL of PR grade Florisil sandwiched between two polyurethane foam (PUF) plugs. The top PUF plug is approximately 50 mm, while the bottom plug is only 25 mm in length. A wire mesh screen at the base of the cartridge supports the PUF plugs and sorbent.

Air flow through the sampler is measured by an in-line venturi magnehelic gauge assembly located downstream of the sampling module which holds the quartz fiber filter. The flow rate is set to approximately 8 theoretical cubic feet per minute at the start of each sampling event by adjusting the sampler's ball-valve assembly until the magnehelic gauge is at the appropriate value. Two simultaneous samplers will be run for 12 hours at the site. The samplers will be calibrated using a GMW Model 40 orifice calibration unit prior to the inception and at the completion of the program.

The PUF plugs and Florisil will be precleaned by Soxhlet extraction with hexane before they are loaded into the glass cartridges. The cartridges will be wrapped in hexane-rinsed aluminum foil and stored in individual hexane-rinsed foam padded glass jars. The filters will be sealed in separate containers.

After sampling, each sample cartridge and filter will be separately rewrapped in hexane-rinsed aluminum foil, then placed together in the original foam padded glass jar container. Containers will be labeled and placed in ice chests maintained at 4°C with "blue ice" packs. A trip blank cartridge and filter will also be rewrapped, placed together in a foam padded glass container, and transported with the samples.

4.4.3.2 Metals

Ambient concentrations of metals upwind and downwind of the site will be measured using a hi-volume sampler. Particulate matter will be collected on 8-inch by 10-inch glass fiber filters and analyzed for metals. The technique will also determine the ambient concentration of particulate matter.

Ambient samples of total suspended particulates (TSP) will be collected using a General Metal Works Model 2000H High Volume Sampler. The filters will undergo analysis for metals following the procedures presented in Section 5. TSP sampling will be conducted in accordance with the Federal Reference Method as described in the EPA Quality Assurance Handbook, Volume II, Section 2.2.11, July 1, 1979.

Because of the high flow rate of the hi-volume sampler, large quantities of particles (up to 1/2 gram) may be collected over a 12-hour sampling period. The ability of this sampler to collect large quantities of particles facilitates laboratory analysis of the filters.

The Model 2000H will be operated at a flow rate of 39-60 cfm, and will be equipped with a Dickinson flow recorder so that accurate flow rate values can be utilized for TSP filter loading calculations.

Specifications for the GMW Model 2000H are as follows:

Sampler Flow Rate:	29-60 scfm
Construction:	Anodized aluminum
Dimensions:	Overall height: 132.1 cm (52 in.)

A schematic diagram of the Model 2000H is shown in Figure 3-14.

EPA recommended procedures for weighing and analyzing TSP and PM-10 Hi-Vol filters will be used. EPA gidelines may be found in 40 CFR, Part 50, Appendix B (7-1-86 Edition).

Each filter will be equilibrated in a controlled humidity chamber at 30% relative humidity before taring. After sample collection and transport to the laboratory, the filter will again be placed in the constant humidity chamber before the final weight is determined. Filters will be weighed on an analytical balance to the nearest milligram. The difference in the weight of the filter before and after sampling (net weight gain) will be the particulate

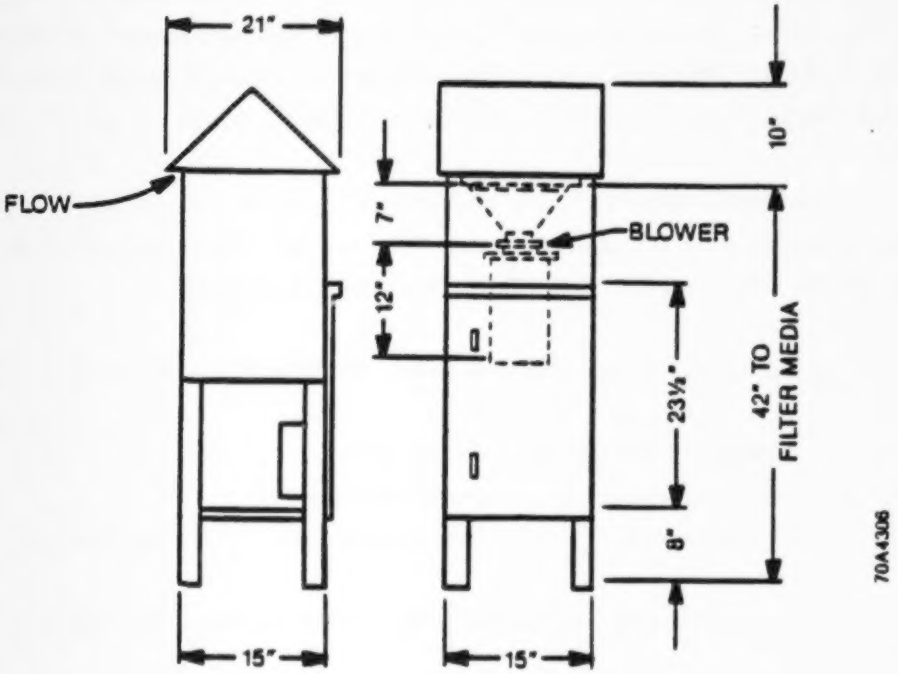

21"
FLOW
15"
7"
12"
BLOWER
10"
23½"
42" TO FILTER MEDIA
8"
15"
70A4306

Figure 4-14. General Metal Works 2000H High-Volume Sampler

loading for that sample. From the weight of particulate on each filter and the volume of air sampled, a concentration in ug/m^3 will be calculated for total suspended particulates in accordance with 40 CFR, Part 50, Appendix B.

4.4.3.3 Meteorological Measurements

During the siting of the ambient air sampling stations, a location for the meteorological monitoring station will also be chosen. The meteorological data will be captured using a battery operated portable meteorological station (Meteorological Research Instruments Model 1072) positioned at the 10-feet level. Wind direction and wind speed will be recorded on a stripchart recorder. Temperature as measured by the meteorological station will be recorded on a stripchart recorder; all other temperature measurements will be recorded by hand and entered in the data sheet.

The following meteorological sensors will be used:

- Temperature--Air temperatures will be recorded using a Fluke® Model 8024B multimeter readout device and thermocouple;
- Wind Speed--Wind speed will be measured using a 3-cup anemometer assembly; and
- Wind Direction--Wind direction will be measured using a light-weight vane rotating on a stainless steel shaft.

These data may be supplmented with data from the nearest National Weather Service Office.

4.5 Calibration Procedures

Information is presented in this section pertaining to the calibration of sampling equipment. Included are descriptions of each procedure or references to applicable standard operating procedures, the frequency of calibrations, and the calibration standards to be used. Calibration frequency of the field sampling equipment is presented in Table 4-5.

4.5.1 Sampling Equipment Calibration Procedures

An important function in maintaining data quality is the checkout and calibration of the source sampling equipment. Using referenced procedures, the equipment will be calibrated prior to field sampling at the Radian laboratories and the results will be properly documented and retained. If a referenced calibration technique for a particular piece of apparatus is not available, then state-of-the-art techniques are used. A discussion of the procedures used to calibrate this equipment is presented below.

4.5.1.1 S-Type Pitot Tube Calibration

The EPA has specified guidelines concerning the construction and geometry of an acceptable S-type pitot tube. If the specified design and construction guidelines are met, a pitot tube coefficient of 0.84 can be used. Information related to the design and construction of the S-type pitot tube is presented in detail in Section 3.1.1 of EPA document 600/4-77-027b. Only S-type pitot tubes meeting the required EPA specifications will be used during this project. Prior to the field sampling, the pitot tubes will be inspected and documented as meeting EPA specifications. An example of the pitot inspection sheet is presented in Figure 4-15.

4.5.1.2 Sampling Nozzle Calibration

EPA Method 5 prescribes the use of stainless steel gooseneck nozzles for isokinetic particulate sampling. Calculation of the isokinetic sampling rate requires that the cross-sectional area of the sampling nozzle be accurately and precisely known. All nozzles used for the EPA Method 5 particulate sampling and Modified Method 5 sampling will be thoroughly cleaned,

TABLE 4-5. CALIBRATION FREQUENCY OF FIELD SAMPLING EQUIPMENT

Sampling Equipment	Calibration Before Sampling	Frequency Daily	After Sampling
S-Type Pitot Tube	*		
Sample Nozzle	*		
Differential Pressure Gauge	*		*
Temperature Measuring Devices	*		
Dry Gas Meters	*		*
Analytical Balance	*	*	
CEMs	*	*	*
PUF	*		*
Hi-Volume Sampler	*		*
Meteorological Equipment	*	*	

PITOT TUBE INSPECTION FORM

Date: ____________________ Operator Initials: ____________________

Pitot Tube I.D. No.: ____________ Length: ____________________

Pitot tube assembly level? ____________ yes ____________ no

Pitot tube openings damaged? ____________ yes (explain below) ______ no

Quick connects attached, leak free? ____________ yes ____________ no

α_1 = __________° α_2 = __________°

β_1 = __________° β_2 = __________°

γ = __________°, θ = __________°, A = ________ cm (in.)

$z = A \sin \gamma$ = ________ cm (in.);

$w = A \sin \theta$ = ________ cm (in.);

P_A = ________________ cm (in.);

P_B = ________________ cm (in.);

D_t = ________________ cm (in.)

Acceptance Criteria:

α_1 and $\alpha_2 < 10°$ __________ yes __________ no

β_1 and $\beta_2 < 5°$ __________ yes __________ no

$z < 0.32$ cm (1/8 in.) ______ yes __________ no

$w < 0.08$ cm (1/32 in.) _____ yes __________ no

P_A and P_B, $1.05\ D_t < P < 1.50\ D_t$ __________ yes __________ no

D_t, 0.48 cm (3/16 in) $\leq D_t \leq$ 0.95 cm (3/8 in) __________ yes ________ no

Pitot Tube Acceptable? __________ yes __________ no

Comments: ____

____

____

Figure 4-15. Example of the Pitot Inspection Sheet

visually inspected, and calibrated according to the procedure outlined in Section 3.4.2 of EPA document 600/4-77-027b. According to this procedure, three measureents of the inside diameter of the nozzle will be made on different diameters. Using a Vernier caliper, measurements will be made to the nearest 0.001 inch. Nozzles will be considered acceptable if the difference between any two measurements is less than 0.004 inches. A nozzle calibration data sheet is shown in Figure 4-16.

4.5.1.3 Differential Pressure Gauge Calibration

Magnehelic® gauges will be used during this project to measure differential and static pressures. In Section 3.1.2 of EPA document 600/4-77-027b, the technique used to calibrate the Magnehelic® is described. This technique requires that pressure readings of the Magnehelic® be compared directly to that of a gauge oil manometer. The Magnehelic® gauges will be calibrated prior to field sampling and checked at a single representative value following the field sampling. An example Magnehelic® gauge calibration form is shown in Figure 4-17.

4.5.1.4 Temperature Measuring Device Calibration

During source sampling, accurate temperature measurements are required. Thermocouple temperature sensors will be calibrated using the procedure described in Section 3.4.2 of EPA document 600/4-77-027b. Each temperature sensor will be calibrated at a minimum of three points over the anticipated range of use (if possible) against a NBS-traceable mercury-in-glass thermometer. All sensors will be calibrated prior to field sampling. Figure 4-18 presents an example calibration form.

4.5.1.5 Dry Gas Meter Calibration

Dry gas meters (DGMs) will be used in the SASS, Modified Method 5, Method 5, and VOST trains to monitor the sampling rate and to measure the sample volume. All dry gas meters will be calibrated (documented correction

Date (DDMMYY): ____________

Initials of Calibrator: ________

Nozzle Identification No.	D_1 **(inches)**	D_2 **(inches)**	D_3 **(inches)**	**Average Diameter (inches)**

NOTE: The **maximum** acceptable difference between any two measurements is 0.004 inches. If this tolerance cannot be met, the nozzle should not be used.

Figure 4-16. Nozzle Calibration Data Sheet

DATE: ______________________ OPERATOR INITIALS: ____________

TYPE OF MAGNEHELIC (CIRCLE ONE): ΔP , ΔH , SPARE

MAGNEHELIC I.D. NO. ____________ RANGE: ______ " H_2O

REFERENCE MANOMETER ΔP_1	MAGNEHELIC ΔP_2	% ERROR $\frac{\Delta P_1 - \Delta P_2}{\Delta P_1} \times 100$
①		
②		
③		
④		
⑤		

DATE: ______________________ OPERATOR INITIALS: ____________

TYPE OF MAGNEHELIC (CIRCLE ONE): ΔP , ΔH , SPARE

MAGNEHELIC I.D. NO. ____________ RANGE: ______ " H_2O

REFERENCE MANOMETER ΔP_1	MAGNEHELIC ΔP_2	% ERROR $\frac{\Delta P - \Delta P_2}{\Delta P_1} \times 100$
①		
②		
③		
④		
⑤		

Figure 4-17. Differential Pressure Gauge Calibration Form

TEMPERATURE INDICATOR IDENTIFICATION NO. (T_{Test})	NBS THERMOMETER SERIAL NO. (T_{STD})	DATE (MMDDYY)	INITIALS OF CALIBRATOR	CALIBRATION DATA	
				MEASURED T_{Test}	MEASURED T_{STD}

Figure 4-13. Temperature Sensor Calibration Form

factor at standard conditions) just prior to the departure of the equipment to the field. A post-test calibration check will be performed as soon as possible after the equipment has been returned to Radian in Austin, Texas. The pre- and post-test calibrations should agree within 5 percent.

The dry gas meters to be used in the SASS, Modified Method 5, and Method 5 trains will be calibrated using the calibration system illustrated in Figure 4-19. Using the procedure outlined in Section 3.3.2 of EPA document 600/4-77-237b, a positive pressure leak-check of the system will be performed prior to calibration. To perform the leak-check, the system is placed under approximately ten inches of water pressure and a gauge oil manometer is used to determine if a pressure decrease can be detected over a one-minute period. If leaks are detected, they will be eliminated before actual calibrations are performed.

To calibrate a dry gas meter, the pump will be allowed to run for 15 minutes after the sampling console is assembled and leak-checked. Once the pump and dry gas meter are warmed up, the valve on the console is adjusted to obtain the desired flow rate. After 10 minutes, the valve is closed and a final set of data is recorded. A duplicate calibration is then performed at the same flow rate. If necessary, additional calibrations are conducted until the calibration results (Y_i) vary by no more than 2 percent. The average Y_i is then calculated and recorded on the face of the DGM console. An example DGM calibration data sheet is presented in Figure 4-20 and a post-test calibration check form is shown in Figure 4-21.

During this project, Rockwell dry gas meters will be used during the SASS, Modified Method 5, and Method 5 tests. The Modified Method 5 and Method 5 DGM calibrations will be performed at Radian's Austin, Texas laboratory using an American Wet Test Meter as an intermediate standard. Using a five-foot American Bell Prover, the intermediate standard is calibrated annually by Carl Poe Co., Inc., in Houston, Texas. The bell jar is routinely calibrated and certified using a "strapping technique" that is traceable to an NBS reference standard. The SASS DGM calibrations will be performed by Fluid Meter Service Corporation using a ten-foot American Bell Prover.

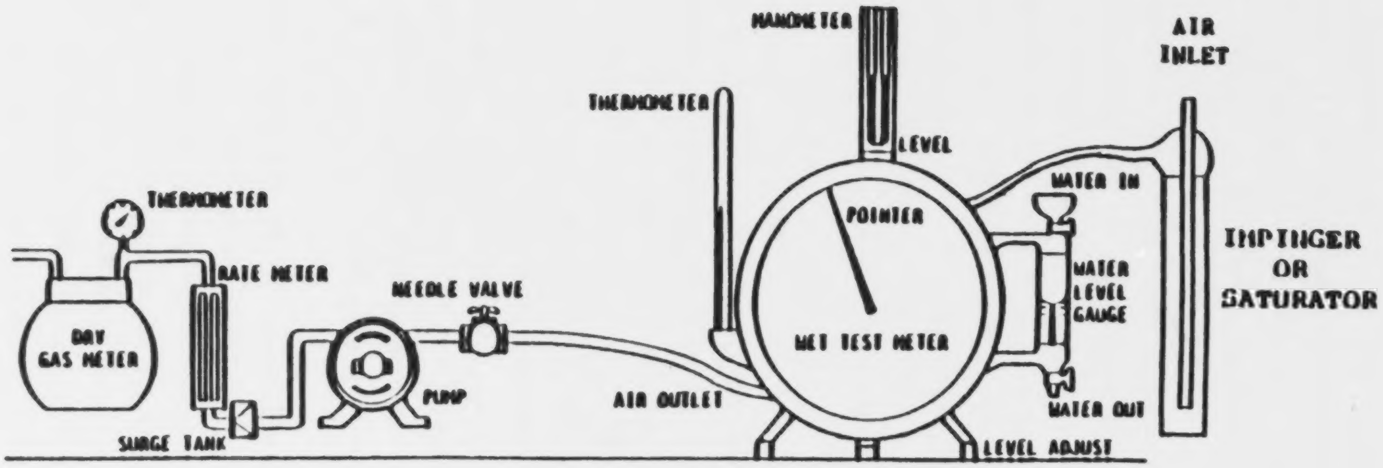

MANOMETER
AIR INLET
THERMOMETER
LEVEL
WATER IN
POINTER
IMPINGER
OR
SATURATOR
THERMOMETER
RATE METER
NEEDLE VALVE
WATER
LEVEL
GAUGE
DRY
GAS METER
WET TEST METER
PUMP
AIR OUTLET
WATER OUT
SURGE TANK
LEVEL ADJUST

Figure 4-19. Dry Gas Master Calibration System

Date ________ Calibrated by ________ Meter or Console No. ________

Barometric pressure, P_b = ________ in. Hg Wet Test Meter No. ________

Test DGM Flow Rate (ACFM)	Orifice Setting (ΔH) (in. H_2O)	Gas Volumes (ft³)						Temperature °F									Elapsed Time (min) (θ)	Vacuum (2-4 in. Hg)
		Wet Test Meter			Dry Gas Meter			Wet Test Meter			Dry Gas Meter							
											Inlet		Outlet					
		Initial	Final	Delta (V_{std})	Initial	Final	Delta (V_{test})	Initial	Final	Avg (T_{std})	Initial	Final	Initial	Final	Avg (T_{test})			

Test DGM Flow Rate (ACFM)	Orifice Setting (ΔH)	$Y_i = \frac{V_{std}\, P_b \left(T_{test} + 460\right)}{V_{test}\left(P_b + \frac{\Delta H}{13.6}\right)\left(T_{std} + 460\right)}$	$\Delta H@_i = \frac{.0317\, \Delta H}{P_b\left(T_{test} + 460\right)}\left[\frac{\left(T_{std} + 460\right)\theta}{V_{std}}\right]^2$
Average		Y =	ΔH@ =

Method 5 Acceptance Criteria

1) $Y_i = Y \pm 0.02Y$
2) $\Delta H@_i = \Delta H@ \pm 0.15\, \Delta H@$ (recommended)
3) $\Delta H@ = 1.84 \pm 0.25$ (recommended)
4) Calibration vacuum maintained at 2-4 in. Hg

Figure 4-20. Dry Gas Meter Calibration Form

Date ____________ Calibrated by ____________ Meter or Console No. ____________

Barometric pressure, P_b = ____________ in. Hg. Wet Test Meter No. ____________

Test DGM Flow Rate (ACFM)	Orifice Setting (ΔH) (in. H_2O)	Gas Volumes (ft³)						Temperature °F								Elapsed Time (min) (θ)	Vacuum (2-4 in. Hg)
		Wet Test Meter			Dry Gas Meter			Wet Test Meter			Dry Gas Meter						
											Inlet		Outlet				
		Initial	Final	Delta (V_{std})	Initial	Final	Delta (V_{test})	Initial	Final	Ave (T_{std})	Initial	Final	Initial	Final	Ave (T_{test})		

Test DGM Flow Rate (ACFM)	Orifice Setting (ΔH)	$Y_i = \frac{V_{std} P_b (T_{test} + 460)}{V_{test} \left(P_b + \frac{\Delta H}{13.6}\right)(T_{std} + 460)}$
Average		Y =

Acceptance Criteria

1) Y = Pretest Y ± 5%

2) Vacuum maintained at ≥ highest vacuum encountered during most recent field testing.

3) $Y_i = Y \pm 0.02\ Y$

Figure 4-21. Dry Gas Meter Post-Test Calibration Check Form

A Singer Model DTM-115 low flow DGM will be used during the VOST testing. The Singer DGM calibrations will be performed using a GCA/Precision Scientific wet test meter. The calibration system and procedure for the Singer DGM is similar to the calibration used for the Rockwell DGM except the Singer DGM will be calibrated at a flowrate of 0.5 to 1.0 liter per minute.

4.5.1.6 Analytical Balance Calibration

During the field measurement program, the analytical balances will be calibrated over the expected range of use with standard weights (NBS Class S traceable) on a daily basis. Measured values must agree within ±0.1 mg.

4.5.1.7 CEM Calibration

CEM calibration is discussed in Section 5.19.7.

4.5.1.8 PUF Sampler Calibration

Calibration of the General Metal Works (GMW) PS-1 sampler is performed using a GMW Model 40 orifice calibration unit (OCU). This unit is initially calibrated by the manufacturer against a standard rootsmeter. The manufacturer's calibration is accepted only if a linear regressions analysis of the data yields a correlation coefficient of at least 0.9998.

The GMW PS-1 samplers will be calibrated prior to inception of the project. The sampler will then be recalibrated at the end of the project. The calibration information is recorded on a standardized data form.

A calibration table with the sampler magnehelic gauge readings and corresponding flow rates is generated. The assumed relationship between these two values is:

$$Y = m X^{.5} + b$$

where, X = magnehelic gauge reading, in inches water
Y = corresponding flow rate, in tcfm
m = linear regression slope
b = linear regression intercept

as long as the resulting correlation coefficient is at least 0.9990. If the regression correlation coefficient derived from the calibration data is less than 0.9990, then the calibration is void and must be redone.

4.5.1.9 Hi-Volume Sampler Calibration

Multipoint flow rate calibrations of the Hi-Vol sampler flow rate recorders are performed using the procedures outlined in 40 CFR, Part 50.11, Appendix B, July 1, 1975. A calibrated orifice and a series of five resistance plates are used to calibrate the flow recorder response.

The following procedures are used to perform the flow rate calibration:

1. The quartz filter is removed and replaced with an adaptor base plate.

2. Set up the water manometer and performa a manometer leak check.

3. Record the chart recorder reading with sampler "off" on the calibration form.

4. Attach the orifice housing and a #13 resistance plate to the sampler. Connect the manometer. Turn the sampler "on" and allow it to run for approximately 5 minutes. Measure the pressure drop across the calibrated orifice using the water

manometer. Record this reading to the nearest 0.05 inch on the calibration form.

5. The flow rate response is measured at four additional points by separately inserting the #18, #10, #7, and #5 resistance plates as indicated in Step 4 (no adjustments are made). Record the indicated chart flow and the manometer reading for each resistance plate on the calibration form.

6. Disconnect the manometer.

7. Check the sampler for air leaks by placing your hand over the orifice inlet momentarily. The chart recorder pen should drop to the zero reading seen in Step 3.

8. Correct any leaks at this time. If any leaks are found or if an adjustment to the flow recorder must be made, a full calibration must be performed after the changes are made. Repeat Steps 3 through 8.

9. Record time, temperature, barometric pressure, and Hi-Vol motor and transducer serial number on the calibration form.

10. Construct a plot of indicated chart flow versus actual flow rate using the chart on the calibration form. If any point deviates from the best fit curve by more than 1 cfm, repeat that plot.

11. Replace the Hi-Vol filter.

12. The sample flow rate through the filter should be between 39-60 ft^3/min (CFM). To determine this, place the orifice kit assembly with no restriction plate over a filter and tighten

the base plate securely. Turn the sampler "on" and record the manometer reading. The flow rate can be calculated using the equation:

Flow Rate (CFM) = Manometer Reading (inches) x Orifice Kit Slop + Orifice Kit Intercept.

The orifice kit flow correction slope and intercept are calculated from the vendor calibration data. This calculation does not encompass temperatures and pressure corrections. Its accuracy is ±15% (±3 cfm).

4.5.1.10 Meteorological Equipment Calibration

The following calibration procedures for the meteorological sensors will be performed once in the field prior to sampling:

- Wind Direction--The wind sensor will be set at north, south, east, and west. The recorder will be checked at each setting for correct response. The setting for north will be determined using a compass. A correction for the site declination will be made during data analysis;
- Wind Speed--The chart speed will be checked for consistent movement; and
- Temperature--The thermocouples will be calibrated by comparison of three separate measurements against an ASTM reference thermocouple at different times of day. The measuring thermouple will be positioned in close proximity to the temperature sensor of the meteorological station. The temperature read-out of the stripchart recorder on the meteorological station will then be compared to the reference thermocouple.

All calibration data and test data from meteorological sensors will be recorded on the meteorological instrumentation calibration form presented as Figure 4-22.

4.6 Sample Custody

Sample custody procedures for this program are based on EPA recommended procedures. Since samples will be analyzed on site, at Radian's permanent laboratory facilities, and at Huffman Labs, the custody procedures discussed below will emphasize careful documentation of monitoring, sample collection, and field analytical data and the use of chain of custody records for samples being transported.

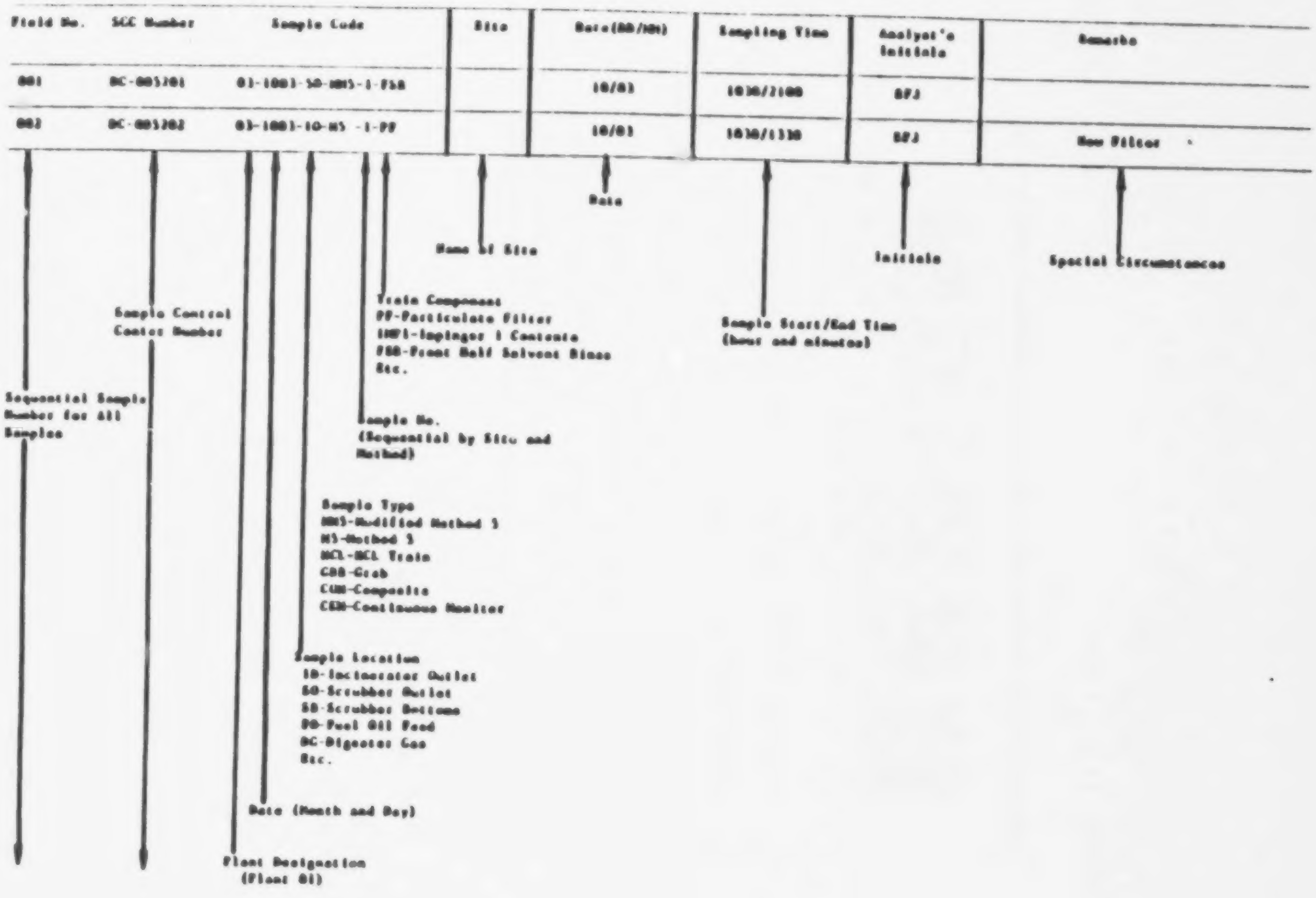
The field sampling leader will be responsible for ensuring that proper custody and documentation procedures are followed for the field sampling and field analytical efforts. He will be assisted in this effort by the sampling personnel involved in sample recovery.

All sampling data, including sampling times, locations, and any specific considerations associated with sample acquisition, will be recorded on preformatted data sheets. Following sample collection, all samples will be logged into a master logbook (bound notebook) and given a unique alphanumeric identification number. Any specific sample preservation, storage, or on-site analysis information will also be noted. An example master logbook format is shown in Figure 4-23. Sample labels (Figure 4-24) and chain of custody seals (Figure 4-25) will be completed and affixed to the sample container. Finally, chain of custody forms (Figure 4-26) will be completed by any personnel involved in the sampling handling.

DATE/ TIME	TEMPERATURE		PERCENT DIFFERENCE	ZERO WIND SPEED READING	ZERO DEGREES WIND DIRECTION READING
	STANDARD	TEST			

METEROLOGICAL INSTRUMENTATION CALIBRATION FORM

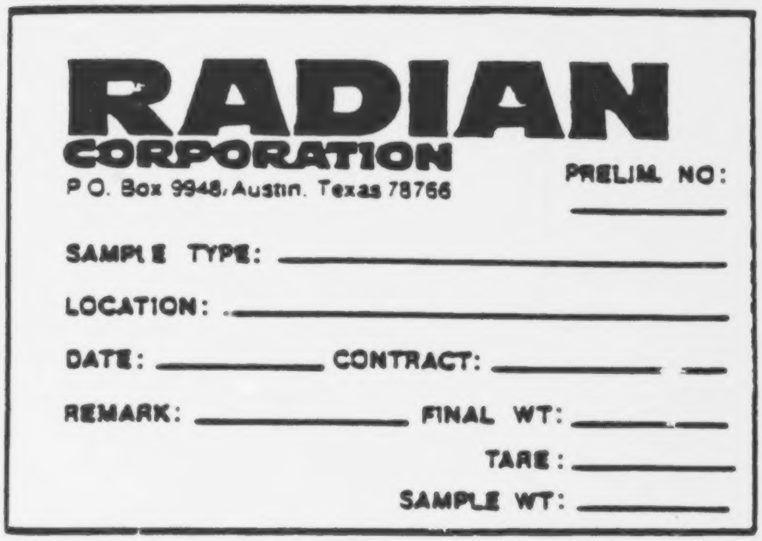
Figure 4-22. Meteorological Instrumentation Calibration Form



Field No.	SCC Number	Sample Code	Site	Date(MM/DD)	Sampling Time	Analyst's Initials	Remarks
001	DC-005301	03-1003-SO-MM5-1-FHR		10/03	1030/2100	BFJ	
002	DC-005302	03-1003-IO-M5 -1-PF		10/03	1030/1330	BFJ	New Filter

Sequential Sample Number for All Samples
Sample Control Center Number
Plant Designation (Plant 03)
Date (Month and Day)
Sample Location
IO-Incinerator Outlet
SO-Scrubber Outlet
SB-Scrubber Bottoms
FO-Fuel Oil Feed
DG-Digester Gas
Etc.
Sample Type
MM5-Modified Method 5
M5-Method 5
HCL-HCL Train
GRB-Grab
CMP-Composite
CEM-Continuous Monitor
Sample No. (Sequential by Site and Method)
Train Component
PF-Particulate Filter
IMP1-Impinger 1 Contents
FHR-Front Half Solvent Rinse
Etc.
Name of Site
Date
Sample Start/End Time (hour and minutes)
Initials
Special Circumstances

Figure 4-23. Example of Radian Master Logbook Format



RADIAN
CORPORATION
P.O. Box 9948, Austin, Texas 78766

PRELIM. NO: ____________

SAMPLE TYPE: ______________________________

LOCATION: ______________________________

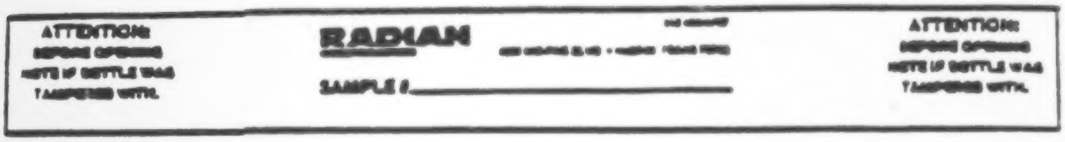
DATE: __________ CONTRACT: __________

REMARK: __________ FINAL WT: __________

TARE: __________

SAMPLE WT: __________

Figure 4-24. Example of Radian Sample Label



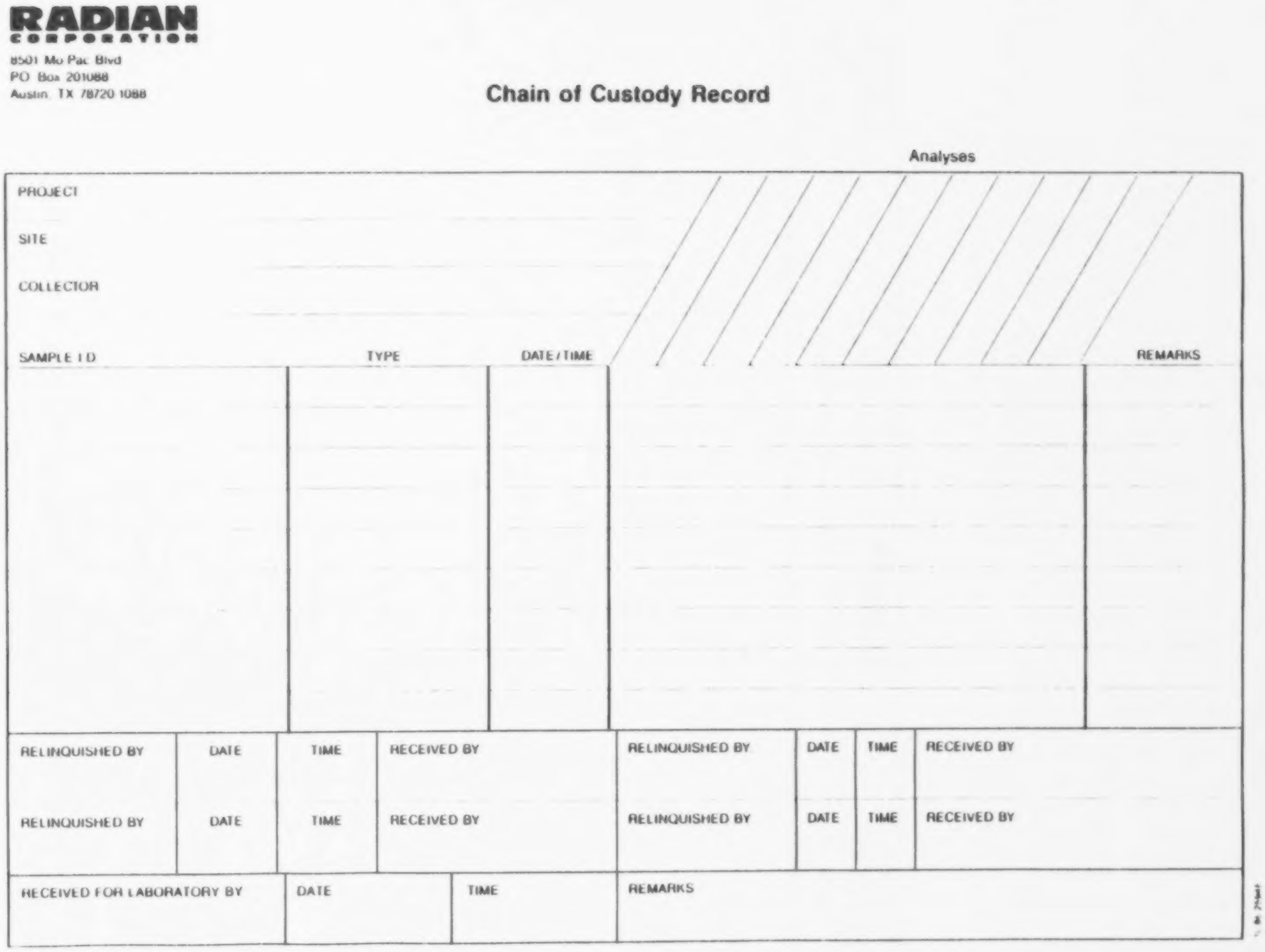
ATTENTION:
BEFORE OPENING
NOTE IF BOTTLE WAS
TAMPERED WITH.

RADIAN

SAMPLE #______________________________

ATTENTION:
BEFORE OPENING
NOTE IF BOTTLE WAS
TAMPERED WITH.

Figure 4-25. Example of Radian Chain of Custody Seal



RADIAN
CORPORATION
8501 Mo Pac Blvd
PO Box 201088
Austin, TX 78720 1088

Chain of Custody Record

Analyses

PROJECT

SITE

COLLECTOR

SAMPLE I D	TYPE	DATE / TIME		REMARKS

RELINQUISHED BY	DATE	TIME	RECEIVED BY	RELINQUISHED BY	DATE	TIME	RECEIVED BY
RELINQUISHED BY	DATE	TIME	RECEIVED BY	RELINQUISHED BY	DATE	TIME	RECEIVED BY

RECEIVED FOR LABORATORY BY	DATE	TIME	REMARKS

Figure 4-26. Example of Radian Chain of Custody Report Form

5.0 LABORATORY PROCEDURES/CALIBRATIONS/QUALITY CONTROL

Samples of solid waste feed, scrubber water, scrubber solids, scrubber make-up water, furnace ash, liquid and solids from flue gas sampling, and samples from ambient air sampling will be analyzed for parameters as specified in Table 4-2. The analytical scheme is discussed briefly below. Descriptions of the analytical methods are presented in the following sections. The preservation requirements and holding times for each type of sample are summarized in Table 5-1.

Solid Streams Analysis

Solid streams including solid waste feed, furnace ash, and scrubber solids will be analyzed for PCBs, PCDD/PCDF, metals, V-PP+10 (volatile priority pollutants +10 next highest peaks), SV-PP+10 (semivolatile priority pollutants +10 next highest peaks), TCLP, EP Toxicity (metals), chlorine, moisture, ash, higher heating value, ultimate analysis, and density. All stream parameters will be measured in triplicate (i.e., one sample from each of three tests) during the test burn and once per test during the parametric testing.

Liquid Streams Analysis

The scrubber liquid samples including scrubber water and scrubber make-up water will be analyzed for PCBs, PCDD/PCDF, pH, chlorine, metals, V-PP+10, SV-PP+10, total organic carbon, total suspended solids, and total dissolved solids. The scrubber effluent liquid will also be analyzed for TCLP and EP Toxicity (metals). Test parameters will be determined in triplicate.

Stack Gas and Primary Furnace Offgas Analysis

Stack gas and primary furnace offgas will be analyzed using continuous emission monitoring systems (CEMs) for carbon monoxide, carbon

TABLE 5-1. PRESERVATION REQUIREMENTS AND HOLDING TIMES FOR SAMPLES

Parameter	Preservation and Storage Requirements	Maximum Holding Time (Days)
pH	-----	6 Hours
EP, TCLP (aqueous)	4°C	Before filtering: Volatiles-14 Semi volatiles-40 Mercury-28 Other metals-180
		After filtering:
	4°C,40 ml,VOA, no headspace	Volatiles-14
	4°C	Semi volatiles-40
	pH<2 with HNO_3	Mercury-28
	pH<2 with HNO_3	Other metals-180
EP, TCLP (non-aqueous)	4°C	Before leaching: Volatiles-14 Semi volatiles-40 Mercury-28 Other metals-180
		After leaching:
	4°C,40 ml,VOA, no headspace	Volatiles-14
	4°C	Semi volatiles-40
	pH<2 with HNO_3	Mercury-28
	ph<2 with HNO_3	Other metals-180
Chloride	None	28
Ultimate	None	None
Ash, Moisture	None	None
HHV	None	None
Density	None	None
TOC	4°C; pH <2 (H_2SO_4)	28
Solids (TSS, TDS)	4°C	7

(Continued)

TABLE 5-1. (Continued)

Parameter	Preservation and Storage Requirements	Maximum Holding Time (Days)
Particulate Matter	None	None
Moisture	None	None
Metals		
Liquid-Dissolved	Filter, pH <2 (HNO_3)	6 Months[a]
Solids	None	6 Months[a]
Soluble Chromium	4°C	ASAP
PCB		
Solids, liquids	4°C	14 (extract), 40 (analyze)
PUF plugs	4°C	72 hrs (extract), 40 (analyze)
PCDD/PCDF	4°C	30
Volatile and Semi-Volatile Organics		
8240	4°C, Dark, Inverted	14
8270	4°C	14 (extract), 40 (analyze)
SASS (PCB, PCDD/PCDF, SV-PP+10)	4°C	30
VOST	4°C	14-42[b]

[a] Except for mercury which is 28 days.

[b] Analysis will be done as quickly as possible.

dioxide, oxygen, nitrogen oxides, and total hydrocarbons. Grab samples will also be collected during each test run and analyzed for carbon dioxide and oxygen using an Orsat analyzer (EPA Method 3). Three Method 5 samples will be collected for analysis of particulate matter, moisture, flow rate, and HCl.

Three integrated sets of VOST samples will be collected for analysis of V-PP+10. Six (6) VOST tube pairs and two condensate vials will be collected for each VOST sample, for a total of 18 tube pairs and 6 condensate vials. Three analyses will result from each set of six VOST tube pairs collected and one condensate vial will be analyzed.

Three Modified Method 5 (MM5) samples will be collected for analysis of soluble chrome.

Three SASS samples will be collected and analyzed for PCBs. In addition, another three SASS samples will be collected and separately analyzed for SV-PP+10 and PCDD/PCDF.

Ambient Air

Ambient air will be sampled daily upwind and downwind of the incineration system. The hi-vol filters will be analyzed for metals, while PUF (polyurethane foam) samples will be analyzed for PCBs. Wind direction and speed and ambient temperature will also be measured.

5.1 Analytical Quality Control Procedures

For each measurement parameter, internal QC procedures are designed to control and assess the performance of the analytical system. The procedures include adherence to accepted laboratory procedures in accordance with the specified analytical protocols, the use of quality control charts, and adherence to acceptance criteria for internal QC sample analyses. A control chart consists of a graphical chart with the vertical scale plotted in units of the test result and the horizontal scale in units of time or sequence of results. The upper and lower control limits shown on the chart are used as criteria for action or for judging the significance of variations between duplicate samples. Daily precision and accuracy data will be plotted by means of these quality control charts to determine if valid, questionable, or invalid data are being generated from day to day.

Internal QC procedures are described in the following sections for each analytical method and are summarized in Table 5-2. They are designed to ensure that the analytical systems remain properly calibrated, free from contamination, and in control. Analyses may not proceed until specific acceptance criteria for calibration, control sample recovery, and blank sample analyses are met. Measurement accuracy for project samples will be determined from matrix spike recoveries. If a matrix spike recovery is unacceptable, a control sample will be analyzed to determine whether the error is due to a problem with the analytical system or due to matrix effects. If the problem is related to the analytical system, the cause of the error will be corrected before analyses proceed; analyses will be repeated to the last acceptable control sample. When matrix spike recovery data indicate that accuracy objectives cannot be met due to matrix interferences, the data will be flagged, and the Project Director will be advised. The feasibility of alternate methods of quantitation (e.g., the method of standard additions) as a means of mitigating the matrix effects will be investigated with the client.

Duplicate matrix spikes will be performed at a rate of one set per twenty samples, minimum or at least one set per matrix.

Acceptance criteria for control sample analyses and drift checks will also be used to control analytical variability, although precision estimates will be based on duplicate matrix spikes. Control sample recoveries will be required to repeat within specified limits or analyses will not proceed. If duplicate analysis results do not agree within the specified acceptance criteria, a third value will be obtained to improve the estimate for precision for that parameter.

TABLE 5-2. SUMMARY OF ANALYTICAL QUALITY CONTROL CHECKS, FREQUENCIES, ACCEPTABLE CRITERIA, AND CORRECTIVE ACTION

Parameter	Analytical Method	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
As Se Hg Pb Tl	SW 7060 7740/7741 7470/7471 7421 7841	Multipoint calibration	Daily prior to analyses	r ≥0.995	Repeat calibration
		Calibration check (single point)	After calibration and after every 10 samples (minimum 2 per set)	Measured value within ±15% of expected value	1) Repeat test 2) Repeat calibration and test 3) See lab manager
		Reagent blank	10%	≤5 x MDL	Will be used to indicate analytical contaminants
		Matrix spike analysis	10%	Recovery within ±20%	1) Repeat test 2) Analyze a QC sample containing each parameter that failed the criteria 3) Flag data
		Matrix spike duplicate analysis	10%	Relative percent difference (RPD) ≤20%	Obtain third value
		LOD check	Quarterly	None	Used to verify current LOD
Metals (Ag, Ba, Be, Cd, Cr, Cu, Ni, Sb, V, Zn)	SW 6010	Mixed standard calibration	Daily	Measured value for high calibration standard ±10% of expected value	Repeat calibration
		Calibration check	10%	Measured value within 10% of true value for element of interest	Repeat calibration
		Reagent blank	10%	≤5 x MDL	1) Repeat test, average results 2) Evaluate system 3) Recalibrate
		Matrix spike analysis	5%	Recovery within ±25% of expected value	Will be used to indicate matrix interference

(continued)

TABLE 5-2. (CONTINUED)

Parameter	Analytical Method	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Metals (continued)		Matrix spike duplicate	5%	Analyses agree within 20% relative percent difference (RPD) for species of interest	Flag data
		ICP intereference check	Run at beginning, middle, and end of daily run	80-120% of true value for EPA check sample elements	1) Repeat calibration 2) See lab manager
		ICP linear range check	Quarterly	Measured value within ±5% of expected value	Tests upper limit of linear range
		LOD check	Quarterly	None	Used to verify current LOD
Total Organic Carbon (TOC)	EPA 415.1	Control Sample	1) Following calibration 2) 10% (minimum 2) 3) Following sample analyses	Recovery between 90-110%	Repeat calibration
		Duplicate analyses	5% (minimum 1)	Relative percent difference (RPD) ≤20%	Repeat sample analyses
		Reagent blank	5% (minimum 1)	None	Will be used to indicate analytical contamination
Dioxins and Furans	SW 8280	Mass scale calibration using PFTBA	Daily prior to sample analyses	Refer to method	Repeat calibration
		Initial calibration Multipoint, 5 levels in triplicate	Prior to method validation, annually, and as required by single point check	Relative standard deviation (RSD) <30%	1) Repeat initial calibration 2) If still unacceptable, make necessary adjustment 3) Repeat initial calibration

(continued)

TABLE 5-2. (CONTINUED)

Parameter	Analytical Method	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Dioxins and Furans (continued)		Single point check	Daily	Agreement within 30% of value predicted from multipoint calibration curve	1) Repeat single point check 2) If still unacceptable, perform new multipoint calibration
		TCDD chromatography check	Daily	25% valley between 1,2,3,4-TCDD and 2,3,7,8-TCDD	Replace column
		PCDD/PCDF retention time window check	Daily unless retention times are stable	Less than ±0.2 min. variation on internal standards	Return retention time standard, adjust SIM windows
		Sensitivity check	Daily	50:1 S/N from 200 ng/mL 2,3,7,8-TCDD standard	Evaluate instrument Take corrective action
		QC sample	Once every project	Refer to method	1) Repeat test 2) Repeat calibration 3) See lab manager
		Extraction blank	5%	None	Used to assess memory effects
		Duplicate analyses	5%	Refer to method	Obtain third value
		Surrogate spike recovery	Every sample	Refer to method	1) Evaluate system 2) Flag data
Volatiles	SW 8240 (GC/MS)	QC check sample	At start of analysis	Refer to method	Correct problem, may require new standard
		Mass scale calibration using PFTBA	Daily prior to sample analyses	Refer to method	Repeat calibration
		Check of mass spectral ion intensities using BFB	Daily prior to sample analyses	Refer to method	Retune instrument Repeat RFB analysis

(continued)

TABLE 5-2. (CONTINUED)

Parameter	Analytical Method	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Volatiles (continued)		Surrogate spikes	Every sample	Refer to method	Flag data
		System performance check compounds	Every 12 hours	RF ≥ 0.300 (0.250 for bromoform)	1) Evaluate system 2) Repeat calibration
		Calibration check compounds	Every 12 hours	% difference <30%	1) Evaluate system 2) Repeat calibration
		Internal standard	All samples	Based on acceptability tests (refer to method)	Flag results as outside data control limits
		Extraction blank	50% per set	None	Used to assess memory effects
		Matrix spikes	5% or per batch <20	Refer to method	1) Run check standard 2) Correct problem 3) Flag data
Semivolatiles	SW 8270	QC check sample	At start of analysis series	Refer to method	Correct problem, may require new standard
		Mass scale calibration with PFTBA	Daily prior to sample analyses	Refer to method	Repeat calibration
		DFTPP relative ion abundance check	Daily prior to sample analyses	Refer to method	Repeat calibration
		Internal standard check	Daily prior to sample analyses	Refer to method	Retune instrument, repeat
		System performance check compounds	Every 12 hours	Refer to method	1) Evaluate system 2) Take corrective action 3) Repeat test 4) See lab manager
		Surrogate spike	All samples	Based on acceptability tests (refer to method)	Flag results as outside data control limits

(continued)

TABLE 5-2. (CONTINUED)

Parameter	Analytical Method	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Semivolatiles (continued)		Extraction blank	Daily prior to sample analyses	Refer to method	1) Clean system 2) Repeat blank analysis
		Matrix spike duplicate samples	5% or per batch <20	Refer to method	1) Run check standard 2) Correct problem 3) Flag data
HCl (Acid Train/ Ion Chromatograph)		Linearity check (multipoint calibration)	Twice (prior to and upon completion of analyses)	Correlation coefficient ≥0.9950 and slope within 10% of running mean for previous six determinations	1) Repeat linearity check 2) Prepare new standards 3) Service instrument
		Duplicate sample analysis	20% of samples	Agreement of analyses 10% CV	1) Repeat analyses
PCBs	EPA 680 (GC/MS)	Mass scale calibration with PFTBA	Daily prior to sample analyses	Refer to method	Repeat calibration
		DFTPP relative ion abundance check	Daily prior to sample analyses	Refer to method	Repeat calibration
		Internal standard check	Daily prior to sample analyses	Refer to method	Retune instrument, repeat
		Surrogate spike	All samples	Based on acceptability tests (refer to method)	Flag results as outside data control limits
		Extraction blank	Daily prior to sample analyses	Refer to method	1) Clean system 2) Repeat blank analysis
		Matrix spike duplicate samples	5% or per batch <20	Refer to method	1) Run check standard 2) Correct problem 3) Flag data

In addition to controlling the quality of data during production, internal QC sample test results will be used to provide estimates of precision and accuracy which will be used in the evaluation and interpretation of project data.

5.2 V-PP+10 Analysis

V-PP+10 analysis will be conducted on several types of samples, including solid, liquid, and gas phase samples. The solid and liquid incinerator samples include:

- Solid Waste Feed;
- Furnace Ash;
- Scrubber Water;
- Scrubber Make-up Water; and
- Scrubber Solids.

The gas phase samples include stack gas and primary furnace offgas samples collected by VOST. V-PP+10 analysis of each type of sample will use the techniques described below.

5.2.1 Stack Gases Analysis for V-PP+10

The Volatile Organic Sampling Train, VOST, will be used to collect samples of the stack gas and primary furnace offgas for quantitation of the V-PP+10. The tenax and tenax/charcoal sorbent traps will be analyzed according to Method 5040 of SW-846, Third Edition, November 1986. This analytical method is based on the quantitative thermal desorption of volatile compounds from the sorbent traps and analysis by purge and trap GC/MS.

According to Method 5040, the contents of the tenax and tenax/charcoal cartridges will be spiked with an internal standard (deuterated benzene), thermally desorbed for 10 minutes at 180°C with organic-free nitrogen or helium gas (at a flow rate of 40 mL/min), bubbled through 5 mL of organic-free water, and trapped on an analytical adsorbent trap. The analytical adsorbent trap will be rapidly heated to 180°C following the 10 minute desorption and the carrier gas flow is reversed so that the effluent flow from the analytical trap is directed into the GC/MS. Volatile compounds are then separated by temperature programmed gas chromatography and detected by low-resolution mass spectrometry as outlined in Method 8240 of SW-846. Method 8420 is discussed in Section 5.2.2 of this document.

Before analysis by GC/MS, all traps, including the blanks, will be spiked with 50 ng of 4-bromofluorobenzene (BFB) standard and the internal standard (deuterated benzene) to provide continuous monitoring of the GC/MS performance relative to BFB.

5.2.1.1 Calibration

Calibration standards will be prepared at a minimum of three concentration levels for each analyte of interest. The calibration standards will be prepared by spiking a blank sorbent trap with a methanolic solution of the calibration standards (including 50 ng of deuterated benzene) using the flash evaporation technique as described in Method 5040. Both tenax and tenax/charcoal traps will be analyzed for each calibration standard. The area response of the characteristic ions of each analyte against the concentration of the internal standards will be tabulated and a response factor for each compound will be calculated. The response factor will be invariant if the response factor value over the working range is a constant (<10% RSD) and the average response factor can be used for calculations. The results can also be used to plot a calibration curve of response ratios versus response factor.

The working calibration curve or response factor will be verified on each working day by the measurement of one or more of the calibration standards. If the response varies by more than ±25% for any analyte, a new calibration standard will be prepared and analyzed for that analyte.

5.2.1.2 Quality Control

Target analytes will be spiked onto the samples to measure recovery efficiency. Blank samples will be analyzed to monitor system contamination. Analysis of duplicate field samples (to ensure against accident, extra field samples are routinely collected and, if unused, available as duplicates) will be performed at a 10% frequency to assess analytical variability.

5.2.2 Liquid and Solid Sample Analysis for V-PP+10

Liquid and solid samples will be analyzed by scanning gas chromatography/mass spectrometry (GC/MS) following SW-846 Method 8240, 3rd ed for the volatile organics shown in Table 3-1. Analyses will be perforemd using a Finnigan MAT model 5100 GC/MS/DS system. Analyte identification and quantitation will be performed using response factors and retention times, relative to the closest eluting of three internal standards, generated from a five point calibration curve. The three internal standards are:

- Bromochloromethane;
- 1,4-Difluorobenzene; and
- Chlorobenzene-d_5.

5.2.2.1 Calibration

The mass spectrometer will be tuned daily to give an acceptable spectrum for bromofluorobenzene (BFB). Relative ion abundance criteria for BFB are shown in Table 5-3.

System performance will be verified initially and after every 12 hours to ensure a minimum average response factor of 0.3 (0.25 for bromoform) for the following system performance check compounds (SPCCs):

- Chloromethane;
- 1,1-Dichloroethane;

TABLE 5-3. BFB* KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5 to 9% of mass 174
176	Greater than 95%, but less than 101% of mass 174
177	5 to 9% of mass 176

*BFB is 4-bromofluorobenzene

- Bromoform;
- 1,1,2,2-Tetrachloroethane; and
- Chlorobenzene.

A five-point calibration, used for generating response factors, will be performed initially using 10, 20, 50, 100, and 200 ug/L standards. As a check on linearity, the relative standard deviation (RSD) must be less than 30 percent for the five response factors calculated for each of the following calibration check compounds (CCCs):

- 1,1-Dichloroethene;
- Chloroform;
- 1,2-Dichloropropane;
- Toluene;
- Ethylbenzene; and
- Vinyl chloride.

A continuing (every 12 hours) calibration check will be performed, following the system performance check, using the CCCs listed above. A single concentration of each CCC will be analyzed and a response factor calculated. The single-point RF for each CCC must be within 25 percent of the average give-point RF. Otherwise, a new five-point calibration must be generated.

5.2.2.2 Quality Control

The quality control procedures for this analytical method are summarized in Table 5-2.

5.3 SV-PP+10 Analysis

SV-PP+10 analysis will be conducted on all sampled streams including:

- Solid Waste Feed;
- Furnace Ash;
- Scrubber Water;
- Scrubber Make-up Water;
- Scrubber Solids;
- Primary Furnace Offgas; and
- Stack Gases.

5.3.1 Stack Gas and Primary Furnace Offgas Analysis for SV-PP+10 Analysis

SV-PP+10 analysis will be performed on the stack gas and primary furnace offgas samples collected using the SASS. The solid (XAD-2 resin) and liquid samples (aqueous condensate) will be prepared for analysis and then analyzed for semivolatile constituents using the procedures outlined in SW-846, "Test Methods for Evaluating Solid Waste," and in the EPA publication, "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater." The SV-PP+10 in the stack gas and primary furnace offgas samples will be analyzed using the procedures specified in the 3rd edition of SW-846 Method 8270.

Note that SV-PP+10 and PCDD/PCDF will be analyzed in the same SASS sample splitting the solvent extract for the two analyses. Surrogates applicable to both analyses will be injected into the samples prior to extraction.

The SASS aqueous condensates will be extracted using SW-846 Method 3520, which is a continuous liquid-liquid extraction. The XAD-2 resin and filter catch of the Modified Method train will be extracted by Soxhlet extraction in accordance with SW-846 Method 3540. Extracts will be concentrated for analysis using a Kuderna-Danish concentration with Snyder columns. The resulting organic extract samples will be diluted in an appropriate solvent as necessary to be within the calibration range of the instrument.

Quantitation will be performed using six internal retention time standards and specific ion response factors generated from the average of a five-point calibration. The six retention time internal standards used for quantitation are:

- Dichlorobenzene-d_4;
- Naphthalene-d_8;
- Acenaphthene-d_{10};
- Phenanthrene-d_{10};
- Chrysene-d_{12}; and
- Perylene-d_{12}.

Target analyte determination will be determined from both retention times and by matching three major ions from the mass spectrum of GC eluent to those in the mass spectral library. A standard solution containing analytes from the target list will be used to generate the mass spectral library.

5.3.1.1 Calibration

The mass spectrometer will be tuned daily to give an acceptable spectrum for DFTPP. DFTPP ion abundance criteria are shown in Table 5-4.

System performance will be verified initilaly and after every 12 hours to ensure a minimum average response factor of 0.050 for the following system performance check compounds (SPCCs):

- N-nitroso-di-n-propylamine;
- Hexachlorocyclopentadiene;
- 2,4-Dichlorophenol; and
- 4-Nitrophenol.

A five-point calibration, used for generating response factors, will be performed initially using 10, 20, 50, 100, and 200 ug/L standards. Specific ion response factors for Method 8270 calibration check compounds must be less than 30 percent RDS over the range calibrated. The CCCs are:

TABLE 5-4. DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[a]

Mass	Ion Abundance Criteria
51	30 to 60% of mass 198
68	Less than 2% of mass 69
70	Less than 2% of mass 69
127	40 to 60% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5 to 9% of mass 198
275	10 to 30% of mass 198
365	Greater than 1% of mass 198
441	Present but less than mass 443
442	Greater than 40% of mass 198
443	17 to 23% of mass 442

[a] Eichelberger, J.W., L.E. Harris, and W.L. Budde. Reference compound to calibrate ion abundance in gas chromatography-mass spectrometry. Analytical Chemistry, 47:995, 1975.

- Phenol;
- 1,4-Dichlorobenzene;
- 2-Nitrophenol;
- 2,4-Dichlorophenol;
- Hexachlorobutadiene;
- 4-Chloro-3-methylphenol;
- Acenaphthene;
- 2,4,6-Trichlorophenol;
- N-nitroso-di-n-phenylamine;
- Pentachlorophenol;
- Fluoroanthene;
- Di-n-octylphthalate; and
- Benzo(a)pyrene.

A continuing (every 12 hours) calibration check will be performed, following the system performance check, using the CCCs listed above. A single concentration of each CCC will be analyzed and a response factor calculated. The single-point RF for each CCC must be within 30 percent of the average five-point RF. Otherwise, a new five-point calibration must be generated.

5.3.1.2 Quality Control

The quality control procedures for the semivolatile analytical method are summarized in Table 5-2.

5.3.2 Liquid and Solid Sample Analysis for SV-PP+10

Samples of the solid waste feed, scrubber water, scrubber make-up water, scrubber solids, and furnace ash will be analyzed by SW-846 Method 8270. Semivolatile compounds analyzed by Method 8270 are listed in Table 3-1. Liquid samples will be extracted using Method 3520, continuous liquid-liquid extraction. Solid samples will be extracted using Method 3540, Soxhlet extraction. Extracts of liquid and solid samples will be analyzed for semivolatile organic contaminants using GC/MS.

5.3.2.1 Calibration

The calibration methods for Method 8270 will follow procedures described in Section 5.3.1.1.

5.3.2.2 Quality Control

Quality control measures will follow those outlined previously in Section 5.3.1.2.

5.4 PCB Analysis

EPA Method 680 will be used to analyze stack gas, primary furnace offgas, ambient air, PUF samples, and solid and liquid samples for PCBs by GC/MS. When analyzing solids, liquids, or other types of extractable samples, the method of choice is generally gas chromatography (GC). The analysis is low in cost and provides a low detection limit. Identification by GC is accomplished by using identifiable patterns given off by the mixture of the various chlorinated isomers of each PCB. Quantitation is achieved by choosing three or more of the larger peaks and developing an average response factor for that particular PCB using the areas from these peaks.

Analysis of filters resulting from flue gas sampling such as those collected at the exhaust stack or primary furnace is much more difficult. Since PCBs contain a variety of chlorinated biphenyls containing two to ten chlorines, the volatility and destruction efficiencies become much more critical. The analysis may be biased toward the more volatile fraction, usually those biphenyls containing two to six chlorines. The result is that a distinctive pattern for a certain PCB is not observed. With the other compounds present in the stack gas causing interference, it is virtually impossible to identify specific PCB isomers by GC analysis.

GC/MS analysis using selected ion monitoring is superior for PCB analysis of gas-derived samples. This type of analysis monitors for ions indicative of biphenyls with two chlorines, three chlorines, etc. The results are usually quantitated chlorinated biphenyl congeners instead of by Aroclor number (1254, 1260, etc.) as is most common.

In this program, all PCB analysis will be determined by GC/MS.

5.4.1 Stack Gas and Primary Furnace Offgas Analysis for PCBs

Samples of the stack gas and primary furnace offgas will be collected using a SASS train with XAD-2 as the adsorbent resin for PCB analysis. The sample is recovered using methanol and methylene chloride. The SASS train will provide three subsamples which will be extracted separately and the extracts combined for analysis. The three subsamples are:

- The glass fiber filter,
- Methanol and methylene chloride rinses of the probe and filter holder, methylene chloride rinse of the condenser/resin trap including the XAD-2,
- Aqueous condensate.

Each subsample will be extracted separately by appropriate SW-846 methods. Sample extracts will then be combined and concentrated to a single final extract.

Sample extract components are separated with capillary column gas chromatography (GC) and identified and measured with low resolution, electron ionization mass spectrometry (MS). The mass spectrometer will be operated in the specific ion monitoring (SIM) mode.

5.4.1.1 Calibration

Four surrogate compounds are added to the three subsamples sample preparation; these compounds are $^{13}CF_{12}$-monochlorobiphenyl, $^{13}C_{12}$-tetrachlorobiphenyl, $^{13}C_{12}$-octachlorobiphenyl, and ^{13}C12-decachlorobiphenyl. Two internal standards, chrysene-d_{12} and phenanthrene-d_{10}, are added to each sample extract before GC/MS analysis and are used to calibrate MS response. Each concentration measurement is based on an integrated ion abundance of one characteristic ion. While acceptable recovery limits for PCBs on XAD have not been established, the guidelines of -50% to +25% from Method 680 will be used as a basis for flagging data.

As previously stated, PCBs will be identified and measured as congener groups (i.e., by level of chlorination). A concentration is measured for each PCB congener group. Total PCB concentration in each sample extract is obtained by summing congener group concentrations. Nine selected PCB congeners are used as calibration standards, and one internal standard, chrysene-d_{12}, is used to calibrate MS response to PCBs, unless sample conditions require the use of the second internal standard, phenanthrene-d_{10}.

5.4.1.2 Quality Control

Quality control measures will follow those outlined on Table 5-2.

5.4.2 Liquid and Solid, and Ambient Air Sample Analysis for PCBs

EPA Method 680 will be followed for analysis of the solid waste feed, furnace ash, scrubber water, scrubber make-up water, scrubber solids, and the ambient air PUF plugs for PCBs. Solid samples and PUF plugs will be Soxhlet extracted using Method 3540 and liquid/liquid extraction by Method 3520 will be used for extraction of liquid samples.

5.4.2.1 Calibration

The calibration methods for Method 680 have been described in Section 5.4.1.1.

5.4.2.2 Quality Control

Section 5.4.1.2 outlines quality control procedures for PCB analysis by Method 680.

5.5 Dioxins and Furans (PCDD/PCDF) Analysis

SW-846 Method 8280 will be used to analyze stack gas, primary furnace offgas, solid and liquid samples for polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) by GC/MS.

Method 8280 is a GC/MS procedure used to determine the concentration of tetra-, penta-, hexa-, hepta-, and octachlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) in a variety of matrices. This procedure uses a matrix-specific extraction, analyte-specific cleanup, and high resolution capillary column gas chromatography/low resolution mass spectroscopy (HRGC/LRMS) techniques.

5.5.1 Stackgas and Primary Furnace Offgas Analysis for PCDD/PCDF

Samples of the stack gas and primary furnace offgas for PCDD/PCDF analysis will be obtained by taking half of the sample extracts from the SASS train samples for SV-PP+10 and PCDD/PCDF (see Section 5.3.1). This extract will be treated to the column cleanup described in SW-846 Method 8280 and then analyzed by GC/MS according to that method. The following surrogates and internal standards are spiked into all samples prior to initial extraction:

- ^{13}C-2,3,7,8-TCDD;
- ^{13}C-2,3,7,8-TCDF;
- ^{13}C-1,2,3,7,8-PeCDD;
- ^{13}C-1,2,3,7,8-PeCDF;
- ^{13}C-1,2,3,6,7,8-HxCDD;
- ^{13}C-1,2,3,4,7,8-HxCDF;
- ^{13}C-1,2,3,4,6,7,8-HpCDD;
- ^{13}C-1,2,3,4,6,7,8-HpCDF;
- ^{13}C-OCDD; and
- ^{13}C-OCDF.

5.5.1.1 Calibration

Twy types of calibration procedures are required. One type, initial calibration, is required before any samples are analyzed and is required intermittently throughout sample analysis as dictated by results of routine calibration procedures. The other type, routine calibration, consists of analyzing the column performance check solution and a concentration calibration solution of 500 ng/mL. The mass spectrometer is tuned daily to give an acceptable spectrum for DFTPP.

Multi-level calibration standards contain the 13 compounds listed in the method. They are prepared at 200, 500, 1,000, 2,000 and 5,000 ng/mL. From injections of the five calibration standards, the relative response factors (RRFs) of analytes are calculated vs. the appropriate internal standards. The relative percent standard deviations (based on triplicate analysis) of the RRFs for each calibration standard will be less than 30 percent or the procedures is repeated.

5.5.1.2 Quality Control

Table 5-2 outlines the quality control acceptance criteria that will be used for this method.

5.5.2 Liquid and Solid Sample Analyses for PCDD/PCDF

SW-846 Method 8280, 3rd ed., will be followed for the analysis of solid waste feed, furnace ash, scrubber make-up water, scrubber water and scrubber solids for PCDD/PCDF.

5.5.2.1 Calibration

The calibration procedures described in 5.5.1.1 will be used for these analyes.

5.5.2.2 Quality Control

Table 5-2 outlines the quality control acceptance criteria that will be used for this method.

5.6 Metals Analysis

Samples of the stack gas and primary furnace offgas particulate matter, ambient air particulate matter, solids, and liquids will be analyzed for metals by inductively coupled argon plasma emission spectroscopy (ICPES) using Method 6010 of "Test Methods for Evaluating Solid Wastes", SW-846, November 1986, third edition and by atomic absorption spectrosocopy (AAS) using Methods 7060, 7421, 7841, 7740, and 7470/7471 of SW-846. The solid samples and particulate matter collected on the filter of the Method 5 train and hi-volume samplers will be solubilized using Method 3050 of SW-846. Prior to analysis liquid samples will be prepared by using Method 3020 from the same publication as the solid samples preparation.

Method 6010 describes the simultaneous, or sequential, determination of elements using inductively coupled plasma atomic emission spectroscopy. The method measures element-emitted light by optical spectrometry. Samples are nebulized, and the resulting aerosol is transported to the plasma torch. Element specific atomic-line emission spectra are produced with are dispersed by a grating spectrometer and monitored for intensity by photomultiplier tubes.

Method 7060, 7041, 7421, and 7740 are graphite furnace atomic absorption techniques for determination of arsenic, antimony lead, and selenium, respectively. Following sample digestion, an aliquot of sample is placed in a graphite tube in the furnace, evaporated to dryness, charred and atomized. The metal atoms to be measured are placed in the light path of an atomic absorption spectrophotometer.

Method 7470/7471 is a cold-vapor atomic absorption procedure for determining the concentration of mercury in waste samples. Sample preparation is specified in the method. Following dissolution, mercury in the sample is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer.

There is concern about the loss of some of the nonvolatile metals (e.g., Hg, As, some Pb compounds) through the Method 5 filter (at 248°F). No promulgated EPA method exists (although there are proposed methods) which quantitatively provides for capture of all the volatile metals. Unusual levels of mercury and arsenic are not expected to be found. In the final report, any limitations on the lead data (or any other elements about which concerns arise) will be noted.

5.6.1 Calibration

Detailed calibration procedures for ICPES systems are described in SW-846, third edition. A response factor is calculated daily for each metal based on three exposures of a calibration standard and calibration blank. The RF is calculaced and stored in the ICPES computer. Following calibration, a mid-level calibration check sample is analyzed; agreement between the measured value and the expected value must be within five percent for analyses to proceed. Calibration is verified by analyzing a QC check standard (prepared independently of calibration standards) every 10 samples; agreement within ±10 percent of the expected value is required for all metals analyzed by ICPES.

The calibration procedures for the graphite furnace and cold vapor AAS systems are described in the respective method in SW-846, third edition. A multi-point calibration curve is generated for each element using a calibration blank and three upscale standards. The correlation coefficient for the linear regression equation must exceed 0.995 to be acceptable. Calibration will be verified every 10 samples by analyzing a QC check sample and calibration blank. Agreement within ±15 percent of the expected value is required; otherwise, a new calibration curve must be generated.

5.6.2 Quality Control

Quality control procedures for metals analyses will include daily multipoint calibration, analysis of QC check samples, duplicate analyses, spiked sample analyses, and blank analyses. Acceptance criteria and corrective action are shown in Table 5-2.

5.7 Soluble Chromium

The stack gas and primary furnace offgas will be sampled in accordance with the method described in Section 4.4.1.3. The resulting samples will be analyzed for soluble chromium (hexavalent chromium) using EPA Method 218.4. By this method, the hexavalent chromium is first chelated using ammonium pyrrolidine dithiocarbamate. The chelated chromium is then extracted from the sample medium using methyl ethyl ketone. The solvent extract is then analyzed by flame atomic absorption spectroscopy.

The stability of hexavalent chromium is not completely understood, and the method recommends that chelation and extraction be carried out as soon as possible. The stack gas and primary furnace offgas samples for soluble chromium analysis will be chelated and extracted on-site soon after sample collection.

5.7.1 Calibration

The atomic absorption spectrometer will be calibrated with a blank and three upscale hexavalent chromium standards. The blank and standards will be chelated and extracted in the same fashion as the samples.

5.7.2 Quality Control

Quality control procedures for the analysis will include multipoint calibration, analysis of QC samples, duplicate analyses, matrix spike duplicates, and analysis of blank 0.1 N NaOH. The matrix spike duplicates will involve spiking of impinger blanks at the sampling location followed, after a representative period of time, by chelation and extraction on-site.

5.8 EP Toxicity

The solid waste feed, furnace ash, scrubber water, and scrubber solids will be analyzed by the RCRA Characteristic of EP Toxicity. The method is prescribed in EPA SW-846 Method 1310 and involves the acidic extraction of solid samples followed by analysis of specific trace metals, pesticides, and herbicides. Liquid samples are analyzed directly. Due to the nature of the wastes to be incinerated, no pesticides or herbicides are expected to be present. The EP Toxicity analysis therefore will be for trace metals only, specifically arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver. The analytical methods for these metals are presented in Table 5-5.

5.8.1 Calibration

Calibration procedures for these methods are described in Section 5.6.1.

5.8.2 Quality Control

Eight metals--arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver--will be analyzed in EP and TCLP extracts to determine if these metals exceed RCRA maximum contaminant levels. Quality control procedures for these analyses will include daily multipoint calibration, analysis of QC check samples, duplicate analyses, spiked sample analyses, and blank analyses. Acceptance criteria and corrective action are shown in Table 5-2.

The method of standard additions (MSA) is required for analysis of all EP extracts. Quality assurance requirements will include a minimum of one blank to be extracted for every 10 extractions that have been conducted in an extraction vessel to determine if memory effects from the extraction equipment are occurring.

TABLE 5-5. ANALYTICAL METHODS FOR EP TOXICITY CHARACTERISTIC METALS

Metal	Digestion Method	Analytical Method	Technique
As	3020	7060	GFAA
Ba	6010	6010	ICP
Cd	6010	6010	ICP
Cr	6010	6010	ICP
Hg	7470	7470	CVAA
Pb	3020	6010	ICP
Se	3020	7740	GFAA
Ag	6010	6010	ICP

GFAA - Graphite Furnace Atomic Absorption.

ICP - Inductively Coupled Argon Plasma Emission Spectroscopy.

CVAA - Cold Vapor Atomic Absorption.

5.9 Toxicity Characteristic Leaching Procedure

The solid waste feed, furnace ash, scrubber water, and scrubber solids will be analyzed by the RCRA Toxic Characteristic Leaching Procedure (TCLP). TCLP is proposed by EPA to expand the Toxicity Characteristic to include additional chemicals and to incorporate a new extraction procedure. The proposal is presented in Federal Register, Volume 51, No. 114, June 13, 1986. The TCLP protocol is described in 40 CFR Appendix II to Part 261. TCLP is applicable to the same eight metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver), four insecticides, and two herbicides as the EP leaching test, but includes 38 additional compounds. The additional organic toxicants represent RCRA Appendix VIII compounds for which there exist promulgated or proposed drinking water standards. The TCLP proposed toxicity characteristic contaminants and regulatory levels are presented in Table 5-6.

Extraction for volatiles involves acidic extraction in a zero-headspace extractor which is rotated in an end-over-end fashion at 30±2 rpm. Extraction for metals and semivolatiles uses the same procedure except that it is done in a glass container rather than the zero-headspace. The metals will be analyzed as described for EP Toxicity. The organic contaminants will be analyzed using SW-846 GC/MS Methods 8240 and 8270.

5.9.1 Calibration

Calibration procedures described in Section 5.2.1.1 and 5.3.1.1 will be used for GC/MS Methods 8240 and 8270. Calibration for metals analysis will be as described in Section 5.5.1.

5.9.2 Quality Control

Quality assurance requirements for TCLP include a minimum of one blank for every 10 extractions that have been conducted in an extraction vessel to determine if memory effects from the extraction equipment are occurring.

TABLE 5-6. PROPOSED TOXICITY CHARACTERISTIC CONTAMINANTS AND REGULATORY LEVELS

Contaminants	Regulatory Level (mg/L)
Acrylonitride	5.0
Arsenic	5.0
Barium	100
Benzene	0.07
bis-(2-Chloroethyl)ether	0.05
Cadmium	1.0
Carbon disulfide	14.4
Carbon tetrachloride	0.07
Chlordane	0.03
Chlorobenzene	1.4
Chloroform	0.07
Chromium	5.0
o-Cresol	10.0
m-Cresol	10.0
p-Cresol	10.0
2,4-D	1.4
1,2-Dichlorobenzene	4.3
1,4-Dichlorobenzene	10.8
1,2-Dichloroethane	0.40
1,2-Dichloroethylene	0.1
2,4-Dinitrotoluene	0.13
Endrin	0.003
Heptachlor (and its hydroxide)	0.001
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.72
Hexachloroethane	4.3

(Continued)

TABLE 5-6. (Continued)

Contaminants	Regulatory Level (mg/1)
Isobutanol	36
Lead	5.0
Lindane	0.06
Mercury	0.2
Methoxychlor	1.4
Methylene chloride	8.6
Methyl ethyl ketone	7.2
Nitrobenzene	0.13
Pentachlorophenol	3.6
Phenol	14.4
Pyridine	5.0
Selenium	1.0
Silver	5.0
1,1,1,2-Tetrachloroethane	10.0
1,1,2,2-Tetrachloroethane	1.3
Tetrachloroethylene	0.1
2,3,4,6-Tetrachlorophenol	1.5
Toluene	14.4
Toxaphene	0.07
1,1,1-Trichloroethane	30
1,1,2-Trichloroethane	1.2
Trichloroethylene	0.07
2,4,5-Trichlorophenol	5.8
2,4,6-Trichlorophenol	0.30
2,4,5-TP (Silvex)	0.14
Vinyl Chloride	0.05

All quality control measures described in the appropriate analytical methods will be followed. The method of standard addition will be used for each waste type if: (1) recovery of the compound from spiked splits of the TCLP extract is not between 50 and 150%, or (2) if the concentration of the constituent measured in the extract is within 20% of the appropriate regulatory threshold. If more than one extraction is being run on samples of the same waste, the method of standard addition need only be applied once and the percentage recoveries applied to the remainder of the extractions.

5.10 Subcontract Analysis

Huffman Laboratories will analyze the solid waste feed, furnace ash, and scrubber solids for chlorine, ash, and ultimate and the solid waste feed for higher heating value.

5.10.1 Chlorine Analysis

Total organic chlorine will be determined in the solid waste feed, furnace ash, and scrubber solids. This analysis will be conducted by Huffman Laboratories, Inc. A total of three possible analytical methods are used. First, because the chlorine concentration can vary considerably depending on the type of waste, the wastes are initially screened using a combustion/absorption/titration technique. The method used is ASTM E442-81. After screening, samples of high concentration are analyzed by combustion/absorption/precipitation as silver chloride. The method used is ASTM D808-81. If during screening the chloride concentration is found to be low, then ion chromatography is the method used for chloride analysis. Ion chromatography is conducted by ASTM D4327-84. The ion chromatography method requires that the sample being analyzed is an aqueous sample. Therefore, when organic liquid, sludge, or solid wastes are analyzed by ion chromatography, the samples must first be combusted using an oxygen bomb (ASTM D808); the combustion gases are absorbed in water and the resulting solution is analyzed by ion chromatography.

5.10.2 Ash Analysis

The ash content of the solid waste feed, furnace ash, and scrubber solids will be determined using ASTM method D482 or D3174. Both methods have basically the same approach. Method D482 is for petroleum products and Method D3174 is for coke or coal-related materials. The sample is ignited and, after burning, is ashed at 775°C in a muffle furnace. The residue is then weighed. Analysis will be performed by the subcontract lab, Huffman Laboratories, Inc.

5.10.3 Ultimate Analysis (C, H, O, S, N, Ash, Moisture)

The solid waste feed will be analyzed for elemental concentrations using Method A003 from "Sampling and Analysis Methods for Hazardous Waste Combustion" for ultimate analysis. The procedure involves the analysis of carbon, hydrogen, nitrogen, phosphorus, sulfur, and chlorine. Oxygen is determined by difference. Method A003 is a conglomerate of ASTM methods. ASTM Method D-3178 analyzes for carbon and hydrogen by burning the samples in a combustion system followed by fixation of the products of combustion in an absorption train for analysis. Nitrogen is analyzed by ASTM Method D3179. The nitrogen in the sample is converted to ammonium salts by destructive digestion. Ammonia is recovered and analyzed titrimetrically. Phosphorus is determined spectroscopically, according to D-2795. ASTM Method D-3177 is used to measure sulfur using bomb calorimetry. The recovered sulfur is precipitated as $BaSO_4$ and determined gravimetrically. Chlorine is determined by Method D-2361 or D-808.

5.10.4 Higher Heating Value Analysis

The higher heating value (HHV) of the solid waste feed fuel will be determined. The heating value is determined using a bomb calorimeter, according to ASTM Method D2015-77 for the solid waste. The HHV analysis will be conducted by Huffman Laboratories, Inc.

5.10.5 Calibration

Calibration procedures of any instruments necessary for the analysis to be performed by Huffman Laboratories will be per laboratory method SOP.

5.10.6 Quality Control

Quality control for samples sent to Huffman Laboratories will be achieved in a variety of ways. In house quality control measures will be relied upon for overall analytical quality. Blind duplicates will be sent for analysis and duplicate analysis will be requested for some samples.

5.11 TSS and TDS Analysis

The concentration of Total Suspended Solids (TSS) and Total Dissolved Solids (TDS) present in the Scrubber Water and Scrubber Make-up Water will be determined using gravimetric procedures specified in the EPA document "Methods for Chemical Analysis of Water and Wastes". TSS and TDS will be determined using Methods 160.1 and 160.2, respectively.

TSS is determined by filtering a known mass of the sample, drying the solids to a constant weight, and using the ratio of the solids mass-to-total sample mass to determine TSS. A drying temperature of 105°C is used. TDS is determined by drying a known mass (volume) of the sample to a constant weight and using the ratio of the solids mass-to-total sample mass to determine TDS.

5.11.1 Calibration

A Mettler Model 4400 top-loader balance that is calibrated semiannually by a manufacturer representative will be used to perform the TSS and TDS analysis. The balance calibration is checked daily against NBS class S weights.

5.11.2 Quality Control

TSS and TDS concentrations obtained by gravimetric methods will be verified by performing replicate analysis on 10% of the samples. An analytical balance capable of weighing to 0.1 mg will be used to quantify solids concentrations.

5.12 pH Analysis

The pH of the scrubber liquor samples will be determined using a portable pH meter and combination electrode with temperature compensation in accordance with EPA Method 150.1. After a sufficient volume of the scrubber liquor is purged through the sample tap, a sample of the scrubber liquor will be collected in a plastic or glass beaker. A portable pH meter and combination electrode will then be used to rapidly measure the pH of the slurry. A mercury thermometer will be used to measure the temperature of the slurry. Both pH and temperature measurements will be recorded on the process log sheet, and the data will be used to help characterize the conditions at which the scrubber system operated during the trial burn.

5.12.1 Calibration

The pH meter should be calibrated with two buffers. One buffer should be at pH 7 and the second should be chosen to cover the range of pH values expected. The temperature compensation control should be set to the buffer temperature.

5.12.2 Quality Control

Quality assurance should include maintaining electrodes properly, using a buffer not used for calibration as a quality control sample, and insuring that the temperature compensation control is set for sample temperature when the measurement is made.

5.13 Density

The bulk density of the solid waste feed, furnace ash, and scrubber solids will be determined using a gravimetric/volumetric method. Sample will be filled to the 250 milliliter mark in a pre-weighed like volume graduated cylinder. The container will be slightly tapped to settle the material, refilling to the mark as necessary. The mass of the 250 milliliter sample will then be determined by weighing and density will be calculated on a mass-per-unit-volume basis.

Density analysis will be determined on the materials "as sampled" with no drying or grinding.

5.13.1 Calibration

Weighings will be performed on a Metler PC4400 toploading balance; it will be calibrated daily with a 100 g class-S weight. The volume of the container will be verified using water and the known density of water.

5.13.2 Quality Control

Replicate analysis will be performed on at least 10 percent of the samples.

5.14 Particulate Matter

Particulates will be measured in the stack gas and primary furnace offgas using EPA Method 5. The acetone wash from the nozzle, probe liner, and glassware prior to the filter on the Method 5 sampling train will be evaporated and the mass determined using an analytical balance. The glass fiber filter will be removed from the Method 5 sampling train, desiccated, and weighed to determine the mass of particulate present on the filter. The weight of the filter and the evaporated wash are totaled and related to the volume of gas sampled to determine the particulate loading.

5.14.1 Calibrations

At the beginning of each day, the analytical balance will be calibrated in the range of use with a set of class-S weights and an internal calibration weight. The internal weight specifications are 100 g ±0.2 mg, adjusted to an apparent density of 8.0 g/cm^3 at an air density of 1200 mg/L.

5.14.2 Quality Control

Prior to sampling, each glass fiber filter will be equilibrated in a desiccator, weighed to determine its initial mass using an analytical balance, and then it will be packaged in a labeled, individual petri dish.

Handling of the filters will be performed in clean areas out of drafts. Tweezers will be used to transfer the filters at all times.

In weighing the filters, both prior to and after sampling, repeat weighings will be performed at $\geq$ six-hour intervals. Repeat weighings must agree within ±0.2 mg to be considered acceptable.

Blank determinations will be performed on each lot of acetone rinse solution. Blank residue must be $\leq$0.01 mg/g or 0.001% of the blank weight.

A field blank of the entire Method 5 train will be obtained, including filter and impinger blanks. The field blank will be prepared by assembling the train as though to collect a sample, but no gas will be pulled through the system. The train will be disassembled and the same procedures used for actual sample recovery will be followed to retrieve the blank.

5.15 Flue Gas Moisture

The moisture content of the gas streams will be determined using the technique specified in EPA Method 4. In this method, a gas sample is extracted at a constant rate from the source and moisture is removed from the sample stream by condensation. The amount of moisture removed is gravimetrically determined and compared to the volume of gas sampled.

Calibration and Quality Control

Each impinger will be weighed to the nearest 0.1 g before and after sampling. Any unusual conditions or occurrences will be noted with each set of samples taken.

5.16 HCl Determination

For the determination of HCl in the stack gas and primary furnace offgas, samples of gas will be passed through a series of impingers immersed in an ice bath. The first two impingers will contain 200 mL of 0.1 N NaOH and will be the Greenburg-Smith type. Following the first two impingers are a dry, modified GreenburgSmith impinger and an impinger containing a desiccant. The aqueous samples from the first two impingers will be analyzed using a Dionex Model 2010 ion chromatograph or similar instrument following Method 27 from the "FGD Chemistry and Analytical Methods Handbook", Radian Corportion, Volume 2, July 1984. This procedure is presented in the Appendix of this document. Calibrations and QA/QC activities from this method will also be followed. In this procedure, the ions in solution are separated on an ion exchange column and detected conductimetrically.

5.16.1 Calibrations

The ion chromatograph will be calibrated for each sample set analyzed. This is accomplished by introducing different concentrations of chloride into the instrument. Calibration standards are prepared from certified standards. A minimum of three concentrations of chloride will be used to generate a calibration curve by the method of least squares fit of data (linear regression). The calibration is considered acceptable if the correlation coefficient is greater than 0.9950.

5.16.2 Ion Chromatograph Quality Control

Quality control will be maintained by analyzing standards, blind duplicates, and QC samples. At least 10% of the samples will be analyzed in duplicate. A detailed description of the IC internal quality control checks and their frequencies are presented in Appendix A.

5.17 Carbon Dioxide

During the proposed sampling for HCl, the collection of CO_2 in the 0.1 N NaOH impinger is a consideration which must be addressed. Because the impinger solutions are caustic, CO_2 will also be removed from the stack gas. Thus, the metered sample gas volume will be low by the amount of CO_2 removed by the impinger solutions. To account for the CO_2 removal, the 0.1 N NaOH impinger solutions will be analyzed for carbonate. The carbonate will be measured on an Oceanography International Corp. Model 524-B carbon analyzer with IR detector using Method 44 from the "FGD Chemistry and Analytical Methods Handbook", Radian Corporation, Volume 2, July 1984. This procedure is presented in Appendix A. Calibrations and QA/QC activities from this method will be followed. Using the carbonate analytical values to determine the amount of CO_2 removed by the impingers, the meter sample gas volume will be corrected.

5.17.1 Calibration

A multipoint calibration curve will be generated for each set of samples as discussed in the procedure presented in Appendix A. A correlation coefficient greater than 0.9950 will be required for analysis to continue.

5.17.2 Quality Control

A QC check sample will be analyzed before and after every 10 field samples analyzed. A method blank will be analyzed to ensure freedom from contamination. One in 10 field samples will be analyzed in duplicate to assess analytical precision. A detailed description of the internal quality control checks an their frequencies are presented in Appendix A.

5.18 Oxygen and Carbon Dioxide Analysis

Grab bag samples will be collected in the field according to EPA Method 3 for CO_2 and O_2. These samples will be analyzed within 3 hours of collection using an Orsat analyzer. The Orsat apparatus measures oxygen and carbon dioxide by selectively absorbing each gas in absorbing solutions.

5.18.1 Calibration

The Orsat is calibrated as received from the manufacturer using a certified gas mixture of O_2 and CO_2 or ambient air. This calibration is performed to verify proper operation of the unit. If the unit is not accurate within ±2% of the calibration gas, then the absorbing solution(s) will be changed and the calibration repeated.

5.18.2 Quality Control

Repeated passes of the sample through each absorbing solution will be made until two consecutive readings are the same or differ by no more than

0.2% for CO_2 and O_2. The Orsat analyzer will be checked for leaks prior to and following these analyses.

5.19 CEMs of CO, CO_2, O_2, NO_x and THC

CEMs will be used during the trial burn program to continuously monitor the concentrations of CO, CO_2, O_2, NO_x, and THC of the stack gas and primary furnace offgas. A brief description of each of the gas phase monitors is provided below.

5.19.1 Carbon Monoxide Analysis

A Bendix Model 85-105CA analyzer will be used to measure CO concentrations in the stack gas and primary furnace offgas. The instrument is a non-dispersive infrared (NDIR) analyzer which measures the concentration of CO by infrared absorption at a characteristic wavelength.

5.19.2 Carbon Dioxide Analysis

To measure the CO_2 concentration in the stack gas and primary furnace offgas, an MSA Model 303 non-dispersive infrared (NDIR) analyzer will be used. This instrument measures the concentration of CO_2 by infrared absorption at a characteristic wavelength.

5.19.3 Oxygen Analysis

A Taylor Model 540A oxygen analyzer will be used to determine O_2 concentrations of the stack gas and primary furnace offgas. The Taylor Model 540A measures oxygen concentrations on the basis of strong paramagnetic properties of O_2 compared to other compounds present in combustion gases. In the presence of a strong magnetic field, O_2 molecules become temporary magnets. The Taylor 540A determines the sample gas O_2 concentration by detecting the displacement torque of the sample gas on a test body in the presence of a magnetic field.

5.19.4 Nitrogen Oxides Analysis

A TECO Model 10 analyzer will be used for NO_x measurement. This instrument determines NO_x concentrations by converting all nitrogen oxides present in the sample to nitric oxide and then reacting the nitric oxide with ozone. The reaction produces a chemiluminescence proportional to the NO_x concentration in the sample. The chemiluminescence is measured using a high-sensitivity photomultiplier.

5.19.5 Total Hydrocarbon Analysis

A Beckman Model 400A will be used to continuously measure the concentration of hydrocarbons present in the flue gas. The analyzer utilizes a hydrogen flame ionization detector. The sensor is a burner where a regulated flow of sample gas passes through a flame sustained by regulated flows of a fuel gas and air. Within the flame, the hydrocarbon components of the sample stream undergo a complex ionization that produces electrons and positive ions. Polarized electrodes collect these ions, causing current to flow through an electronic measuring circuit. The ionization current is proportional to the rate at which carbon atoms enter the burner and is, therefore, a measure of the concentration of hydrocarbons in the original sample.

5.19.6 CEMs Calibrations

Calibrations of all continuous monitors will be accomplished by introducing standard gases at the front end of the CEM sampling probe prior to and after daily sampling to allow for the assessment of any impact caused by the sample gas conditioning system, including the heat traced sample line, on the pollutants being monitored. Table 5-7 presents the calibration standard gases which will be used to calibrate the instruments. All instruments will

TABLE 5-7. STANDARDS FOR CONTINUOUS MONITORS

Analytical Parameter	Standard	Source	Documentation
$CO/CO_2/O_2$ Quantitation	CO-5, 400 ppmv, N_2-Balance;	Scott Specialty Gases	Certified*
	CO_2-15%, N_2-Balance;		
	O_2-12%, N_2-Balance;		
	CO-300 ppmv, CO_2-10%, O_2-9%, N_2-Balance (Audit Gas)		
	CO-200 ppmv, CO_2-5%,		
	O_2-3%, N_2-Balance		
NO_x Quantitation	NO-50 ppmv, N_2-balance	Scott Speciality Gases	Certified*
	NO-500 ppmv, N_2-balance		
	NO-250 ppmv, N_2-balance (Audit Gas)		
THC Quantitation	Hexane-50 ppmv, N_2-Balance;	Scott Specialty Gases	Certified*
	Hexane-5 ppmv, N2-Balance		
	Hexane-30 ppmv, N_2-balance (Audit Gas)		

* "Certified" standards are mixtures analyzed by the gas manufacturer with the analytical accuracy certified to be ±2%. Analyses are referenced to NBS standard reference materials, if SRMs exist, or to gravimetric standards, which are gravimetrically traceable to NBS.

undergo multipoint linearity checks (two points plus zero) bracketing the predicted sample values. These checks will be performed at the beginning and end of the sampling program. In all cases, the acceptance criterion for the linearity checks will be a correlation coefficient (r) of $\geq$ 0.9950. If this criterion is not met, the linearity check will be repeated (following instrument maintenance, if necessary) until r $\geq$ 0.9950 is achieved.

An analytical blank and a single-point response factor (RF) standard will be analyzed daily prior to testing for all continuous monitors. The acceptance criterion for the single-point RF will be an RF agreement within 20% of the previous multipoint RF. If this criterion is not met, the multipoint RF check will be repeated. The acceptance criterion for the analytical blank will be $\leq$1% of the instrument span. Drift checks will be performed prior to any calibration or zero adjustment.

5.19.7 CEMs Quality Control

During the testing program, CEMs operation will include the following QC procedures.

A single-point drift check will be performed by analyzing standard used for the single-point RF determination at the end of each day of testing. The acceptance criterion for this analysis will be agreement within 10% of the single-point RF determination. If this criterion is not met, the end of testing RF and the initial single-point RF will be used to quantitate samples assuming a linear drift throughout the test day. Calibration requirements for the continuous monitors are shown in Table 5-8.

Each day each monitor will be challenged with a mid-range certified gas. Reference to these gases will define accuracy and serve as a daily audit for each instrument. Table 5-7 specifies the audit standards as well as the calibration standards.

TABLE 5-8. SUMMARY OF CONTINUOUS MONITOR CALIBRATION REQUIREMENTS

Parameter	Method of Analysis	Type of Calibration	Calibration Standards	Frequency	Acceptance Criteria
CO	Bendix Model 85-105CA (NDIR)	Multipoint (Zero plus 2 upscale)	CO and CO_2 in N_2	Once at site prior to testing	Correlation coefficient $\geq$ 0.9950
		Single point RF	CO and CO_2 in N_2	Daily (prior to testing)	Agreement within 20% of multi-point RF
		Analytical blank	Zero N_2	Daily (prior to testing)	Zero value $\leq$ 1% of span
		Single point drift check	CO and CO_2 in N_2	Daily (at conclusion of analysis)	RF agreement within 10% of RF for single point RF check
CO_2	MSA Model 303 (NDIR)	Same as CO	Same as CO	Same as CO	Same as CO
O_2	Taylor Model 540A (Paramagnetic)	Same as CO	O_2 in N_2	Same as CO	Same as CO
NO_x	TECO Model 10 (Chemiluminescence)	Same as CO	NO in N_2	Same as CO	Same as CO
THC	Beckman Model 400A (Flame Ionization)	Same as CO	Hexane in N_2	Same as CO	Same as CO

5.20 Total Organic Carbon

Total carbon is measured using a carbonaceous analyzer by quantitatively converting the organic and inorganic carbon in a sample to carbon dioxide which is measured by an infrared detector. Total inorganic carbon is determined by sparging carbonates from the sample as CO_2 which is measured by IR. The UV-catalyzed oxidation of organics is not used. Total organic carbon is calculated from the difference of total carbon - total inorganic carbon.

5.20.1 Calibration

A multipoint calibration curve will be generated daily, prior to sample analysis, using a blank and a minimum of three upscale standards. Potassium hydrogen phthalate will be used for total carbon measurements; sodium carbonate/bicarbonate will be used for inorganic carbon measurments. The correlation coefficient for the calibration equation must exceed 0.995.

5.20.2 Quality Control

A QC check sample will be analyzed after every 10 samples; recovery must be within ±10 percent of the expected value.

6.0 DATA REDUCTION, VALIDATION, AND REPORTING

The overall data reduction, validation, and reporting flow scheme for this project is presented in Figure 6-1. Details of data reduction, validation, and reporting are discussed in the remainder of this section.

6.1 Calculations

The following sections detail the calculations which will be performed for data preservation.

6.1.1 PCB, PCDD/PCDF, V-PP+10, SV-PP+10, and Metals Mass Emission Rate Calculations

The MER mass emission rates (MER, ug/hr), for PCB, PCDD/PCDF, V-PP+10, SV-PP+10, and metals in the stack gas and primary furnace offgas samples will be calculated as follows:

$$MER = (M_n)\ (Qsd)/V_d \times 60\ (min/hr)$$

where: M_n = mass of compound collected (ug)

Q_{sd} = dry volumetric flow rate at standard conditions (dscfm)

V_d = dry gas volume sampled at standard conditions (dscf)

6.1.2 Particulate Mass Emission Calculation

The mass emission rate (grains per dscf) will be determined for total particulates based on EPA Method 5 as follows:

$$C_s = (M_n)\ (0.001)\ (15.43)/(V_d)$$

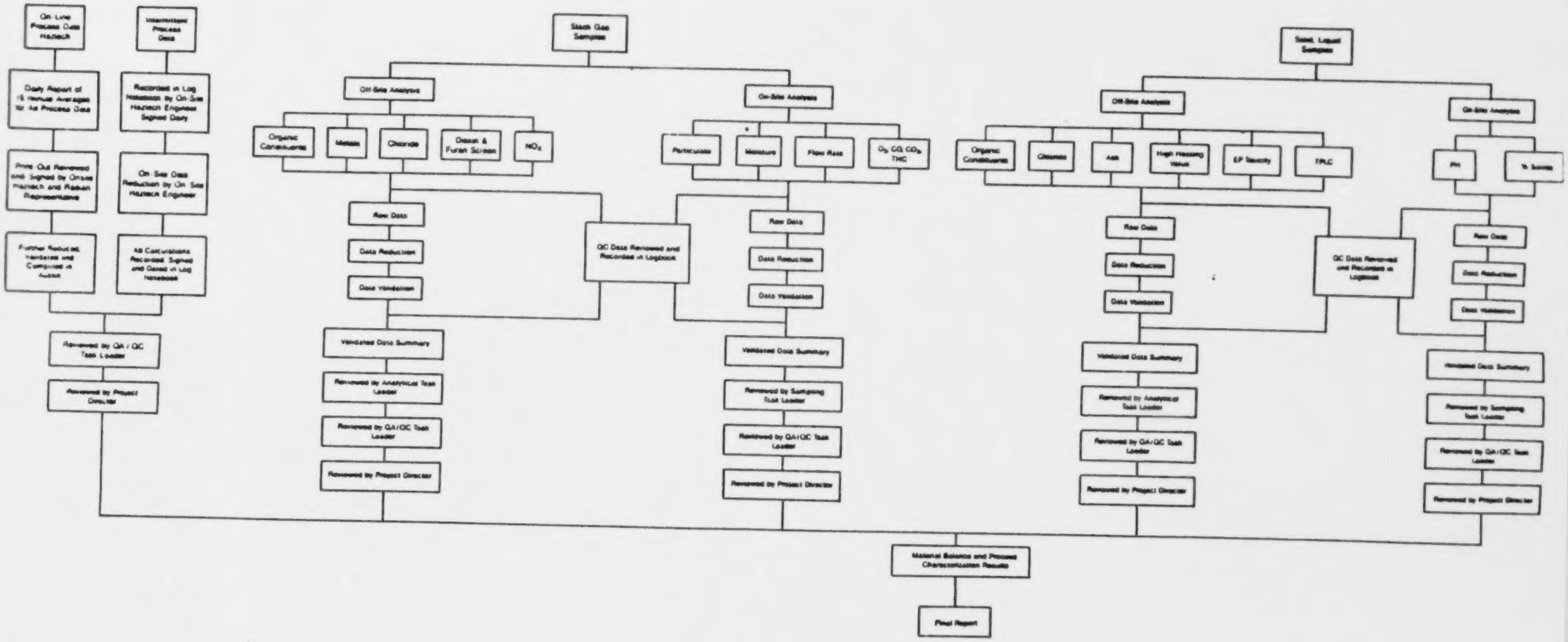

On-Line Process Data Haztech
Intermittent Process Data
Daily Report of 15 minute Averages for All Process Data
Recorded in Log Notebook by On-Site Haztech Engineer Signed Daily
Print-Out Reviewed and Signed by Onsite Haztech and Radian Representative
On-Site Data Reduction by On-Site Haztech Engineer
Further Reduced, Validated and Compiled in Austin
All Calculations Recorded, Signed and Dated in Log Notebook
Reviewed by QA/QC Task Leader
Reviewed by Project Director
Stack Gas Samples
Off-Site Analysis
Organic Constituents
Metals
Chloride
Dioxin & Furan Screen
NO_x
Raw Data
Data Reduction
Data Validation
Validated Data Summary
Reviewed by Analytical Task Leader
Reviewed by QA/QC Task Leader
Reviewed by Project Director
QC Data Reviewed and Recorded in Logbook
On-Site Analysis
Particulate
Moisture
Flow Rate
O_2, CO, CO_2, THC
Raw Data
Data Reduction
Data Validation
Validated Data Summary
Reviewed by Sampling Task Leader
Reviewed by QA/QC Task Leader
Reviewed by Project Director
Solid, Liquid Samples
Off-Site Analysis
Organic Constituents
Chlorides
Ash
High Heating Value
EP Toxicity
TPLC
Raw Data
Data Reduction
Data Validation
Validated Data Summary
Reviewed by Analytical Task Leader
Reviewed by QA/QC Task Leader
Reviewed by Project Director
QC Data Reviewed and Recorded in Logbook
On-Site Analysis
PH
% Solids
Raw Data
Data Reduction
Data Validation
Validated Data Summary
Reviewed by Sampling Task Leader
Reviewed by QA/QC Task Leader
Reviewed by Project Director
Material Balance and Process Characterization Results
Final Report

Figure 6-1. Overall Data Reduction, Validation, and Reporting Flow Scheme

where: C_s = concentration of particulates in the flue gas at dry standard conditions (grains/dscf),

M_n = mass of specie collected (mg),

V_d = dry gas volume sampled at standard conditions (dscf),

0.001 = conversion of milligrams to grams, and

15.43 = conversion of grams to grains.

6.1.3 Chloride Mass Emission Rate Calculations

Scrubber efficiency (SE), used to determine chloride species removal, will be calculated as follows:

$$SE = \frac{Cl_{in} - Cl_{out}}{Cl_{in}} \times 100\%$$

where: Cl_{in} = mass feed rate of chlorine entering the incinerator, and

Cl_{out} = mass emission rate of chlorine (as hydrogen chloride) in the scrubber exhaust gas prior to emission to the atmosphere.

6.1.4 Stack Gas and Primary Furnace Offgas Volumetric Flow Rate

The stack gas and primary furnace offgas volumetric flow rate will be determined on several bases.

6.1.4.1 Stack Gas Velocity Calculation

The velocity of the stack gases based on EPA Method 2 will be calculated as:

$$V_s = 85.48\ C_p \left(\frac{(T_s)(\Delta P)}{(P_s)(M_w)}\right)^{1/2}$$

where: pitot tube constant = $85.48 \frac{ft}{sec} \left(\frac{(lb/lb\text{-}mole)(in.\ Hg)}{(°R)(in.\ H_2O)} \right)^{1/2}$

V_s = velocity of the flue gas (ft/sec),

ΔP = average differential pressure measured by S-type pitot (in. H_2O),

T_s = average gas temperature in duct (°R),

P_s = absolute duct pressure (in. Hg),

M_w = wet gas molecular weight (lb/lb-mole), and

C_p = S-type pitot correction factor (normally 0.84).

6.1.4.2 Gas Volumetric Flow Rate Calculation - Actual Conditions

The volumetric flow rate of the stack gas and primary furnace offgas based on EPA Method 2 will be calculated as:

$$Q_{ac} = 60\ (V_s)(A)$$

where: Q_{ac} = actual volumetric flow rate (acfm),
V_s = velocity of the flue gas (ft/sec),
A = cross-sectional area of the duct (ft_2), and
60 = conversion from seconds to minutes.

6.1.4.3 Gas Volumetric Flow Rate Calculation - Dry Standard Conditions

The volumetric flow rate of the stack gas and primary furnace offgas at dry standard conditions based on EPA Method 2 will be calculated as:

$$Q_{sd} = (1 - B_w)(Q_{ac})(528/T_s)(P_s/29.92)$$

where: Q_{sd} = dry volumetric flow rate at standard conditions (dscfm),

Q_{ac} = actual volumetric flow rate (acfm),

B_w = moisture fraction,

528 = standard temperature (°R),

T_s = average gas temperature in duct (°R),

29.92 = standard pressure (in. Hg), and

P_s = absolute duct pressure (in. Hg).

6.1.5 CO, CO_2, O_2, NO_x, and THC Concentration Calculations

Response factors (RF) for the continuous monitors will be calculated from the single point RF check and the instrument zero value as follows:

$$RF = \frac{C_{std}}{[(\%\ FS)_{std} - (\%\ FS)_{zero}]}$$

Using this response factor, the sample concentration will be calculated using the following equation:

$$C_{smpl} = [(\%\ FS)_{smpl} - (\%\ FS)_{zero}] \times RF$$

where: RF = response factor or calibration factor for the parameter calibrated, CO, CO_2, O_2, NO_x, or THC.

C_{std} = certified concentration of the parameter in the calibration gas,

$(\%\ FS)_{std}$ = calibration standard response expressed as percent of full-scale output (on strip chart recorder or instrument output voltage),

$(\%\ FS)_{std}$ = zero standard response expressed as percent of full-scale output, and

$(\% \ FS)_{smpl}$ = sample response expressed as percent of full-scale output.

6.1.6 Waste Feed Rate Calculations

The concentration of PCB, volatile and semi-volatile priority pollutants, and metals will be reported in units of micrograms per gram for the waste feed solids. These values will be reported for each composite sample for each run. The total mass rate of the individual compounds fed to the incinerator during each burn will be calculated from these concentrations and the process operation data for the amount of each feed material fed to the incinerator during each burn.

6.1.7 Solids/Liquids Analysis Calculations

The concentration of PCB, volatile and semi-volatile priority pollutants, and metals will be reported in units of microgram per liter for the liquid samples and in units of microgram per gram for the solid samples for each test.

6.1.8 Mass Balance

The concentration of chlorine and metals in the waste feed, liquids, solid samples and stack gas and primary furnace offgas emissions will be determined. Mass balances for the compounds will be evaluated as follows:

$$W_{in} + S_{in} = SG_{out} + S_{out} + A_{out}$$

where: W_{in} = compound in waste feed in,

S_{in} = compound in liquid in,

SG_{out} = compound in stack gas or primary furnace offgas emission,

S_{out} = compound in liquids out, and

A_{out} = compound in solid samples out.

6.2 Data Validation

All measurement data will be validated based upon representative process conditions during sampling or testing, acceptable sample collection and testing procedures, consistency with expected and/or other results and adherence to prescribed QC procedures. Any suspect data will be flagged and identified with respect to the nature of the problem with validity. Suspected outliers will be tested using the Dixon Criteria at the five percent significance level.

6.3 Reporting

Reporting responsibilities for this project are shown in Table 6-1.

Radian will evaluate the results of the demonstration test for completeness and representativeness and will perform the calculations for chlorine removal, particulate matter concentrations, emission rates, and other required data. A report of the results of the demonstration test will be submitted to Enviresponse within 90 days after completion of sampling. Data and results interpretation will be presented as necessary in the report. The report will also detail sampling and analytical procedures used during the demonstration test program.

TABLE 6-1. SUMMARY OF DATA REDUCTION, REVIEW, AND VALIDATION AND REPORTING RESPONSIBILITIES

	Personnel Responsibilities		
Task	Data Reduction	Data Review and Validation	Reporting
Quality Assurance Project Plan		D.L. Lewis	M.R. Fuchs
Site-Specific Data Summaries	Field Team Members	M.J. Krall	L.O. Edwards
Analytical Results	S.K. Mertens	M.J. Krall	L.O. Edwards
Final Concentrations, etc.	M.J. Krall	L.O. Edwards	L.O. Edwards

7.0 INTERNAL QUALITY CONTROL CHECKS

Specific QC procedures will be followed to ensure the continuous production of useful and valid data throughout the course of the Shirco Portable Pilot Test Unit demonstration test. The QC checks and procedures described in this section represent an integral part of the overall sampling and analytical scheme. Strict adherence to prescribed procedures is quite often the most applicable QC check. A discussion of both the sampling and analytical QC checks that will be utilized during this program is presented below.

7.1 Sampling Quality Control Checks

Prior to actual sampling on site, all of the applicable sampling equipment will be thoroughly checked to ensure that each component is clean and operable. Each of the equipment calibration data forms will be reviewed for completeness and adequacy to ensure the acceptability of the equipment. Each component of the various sampling systems will be carefully packaged for shipment. Upon arrival on site, the equipment will be unloaded, inspected for possible damage, and then assembled for use.

The following QC checks are applicable to each of the EPA Methods 2, 3, 4, and 5, Modified Method 5, SASS, VOST, metals, and HCl trains.

1. Each sampling train will be visually inspected for proper assembly before every use.
2. All sampling data will be recorded on standard data forms which will serve as pre-test checklist.
3. The oil manometer or Magnehelic® gauge to be used to indicate the differential pressure (P) across the S-type pitot tube will be leveled and zeroed.
4. The number and location of the sampling traverse points will be checked before taking measurements.

5. The temperature measurement system will be visually checked for damage and operability by measuring the ambient temperature prior to each traverse.
6. Duplicate readings of temperature and differential pressure will be taken at each traverse point.
7. All sampling data and calculations will be recorded on preformatted data sheets.

In addition to the general QC procedures listed above, QC procedures specific to each sampling method will also be incorporated into the sampling scheme. These method specific procedures are discussed below.

7.1.1 Sampling Quality Control Procedures for SASS, Modified Method 5, Method 5 and VOST

Samples for PCB, PCDD/PCDF, and semivolatile organics analyses will be collected according to the SASS protocol. Total particulate mass, metals, and chloride species concentrations in the stack gas and primary furnace offgas will be determined using EPA Method 5. Volatile organics will be determined using VOST. Soluble chrome will be determined using a version of the Modified Method 5 train. Quality control for sampling will focus on the following as appropriate for the individual sampling trains.

1. Prior to sampling, each SASS and M5 filter will be placed in a labeled, individual precleaned glass or plastic petri dish.
2. For SASS and VOST sampling, all cleaned glassware, hardware, and prepared sorbent traps will be kept closed with ground glass caps or precleaned foil until assembly of the sample train in the field. The sorbent traps will be immediately recapped when the train is disassembled.
3. Assembly and recovery of the sample trains will be performed in an environment free from uncontrolled dust.
4. For SASS and VOST sampling, sample train blanks will be collected.
5. Prior to sampling, calculations will be made to determine the proper size nozzle required to attain isokinetic sampling.

6. The sampling nozzle will be visually inspected before and after each run for damage.

7. The S-type pitot tube will be visually inspected before and after each run for damage.

8. Each leg of the S-type pitot tube will be leak-checked before and after each run.

9. The oil manometer or Magnehelic® gauge to be used to indicate the differential pressure (P) across the S-type pitot tube will be leveled and zeroed.

10. The number and location of the sampling points will be checked before taking measurements.

11. The temperature measurement system will be visually checked for damage and operability.

12. During sampling, the roll and pitch axis of the S-type pitot tube and the sampling nozzle will be properly maintained.

13. The entire sampling train will be leak-checked before and after each run. If the sampling train is moved from one sampling port to another during a run, the train will be leak-checked before and after the move.

14. Additional leak checks will be performed if the sampling time exceeded four hours.

15. The filter and sorbent trap will be maintained at the proper temperature throughout the test run.

16. Ice will be maintained in the ice bath throughout each run.

17. Dry gas meter readings, P and H readings, temperature readings, and pump vacuum readings will be properly made during sampling at each traverse point.

18. Isokinetic sampling will be maintained within ±10%.

19. In weighing the filters, both prior to and after sampling, repeat weighings will be performed ≥ six hours after the initial weighings. Repeat weighings must agree within ±0.2 mg to be considered acceptable.

20. For M5 sampling, blank determinations will be performed on each lot of acetone rinse solution. Blank residues must be ≤0.01 mg/g or 0.001% of the blank weight.

7.1.2 Quality Control Procedures for Velocity/Volumetric Flow Rate Determinations

Data required to determine the volumetric gas flow rate will be collected using the methodology specified in EPA Method 2. Quality control will focus on the following procedures.

1. The S-type pitot tube will be visually inspected before and after sampling.
2. Both the low-pressure and high-pressure legs of the S-type pitot tube will be leak-checked before and after sampling.
3. The oil manometer or Magnehelic® gauge to be used to indicate the differential pressure (P) across the S-type pitot tube will be leveled and zeroed.
4. The number and location of the sampling traverse points will be checked before taking measurements.
5. The temperature measurement system will be visually checked for damage and operability by measuring the ambient temperature prior to each traverse.
6. All sampling data and calculations will be recorded on preformatted data sheets.

7.1.3 Sampling Quality Control Procedures for Moisture Determination

The moisture content of the gas streams will be determined using the technique specified in EPA Method 4. The following internal QC checks will be performed as part of the moisture determinations.

1. Each impinger will be weighed to the nearest 0.1 grams before and after sampling.
2. The sampling train, including impingers, will be leak-checked before and after each run.
3. Ice will be maintained in the ice bath throughout the run.
4. Any unusual conditions or occurrences will be noted during each run on the appropriate data form.

5. The field sampling team leader will review sampling data sheets daily during testing.

7.1.4 Quality Control Procedures for Molecular Weight Determinations

Samples to be used for determination of stack gas and primary furnace offgas molecular weight will be collected using the grab sampling technique specified in EPA Method 3. Quality control for the Method 3 sampling will focus on the following.

1. The sampling probe will be stainless steel and equipped with a filter (glass wool plug) to remove particulate matter.
2. The sampling train will be purged prior to sample collection.
3. The sampling port will be properly sealed to prevent air in-leakage.

7.1.5 Quality Control Procedures for the HCl Trains

The sample to be used for determining HCl concentrations in the stack gas and primary furnace offgas will be obtained using a modified sampling train. Quality control will focus on the following.

1. Prior to sampling, the impinger trains will be properly assembled for use. The first and second impingers will contain 0.1 N NaOH solution. The third impinger will be dry, while the last impinger will contain silica gel.
2. The entire sampling train will be leak-checked before and after each run.
3. The probe and sample line prior to the impinger train will be maintained above the dew point of the sample gas.
4. Ice will be maintained in the ice bath throughout each run.
5. Dry gas meter readings will be properly made at the start and end of sampling.
6. A constant (i.e., within 10%) sampling rate will be maintained.

7.2 Analytical Quality Control Procedures

All analyses for this program will be performed using accepted laboratory procedures in accordance with the specified analytical protocols. Gas standards used for quantitation will be certified (± 2% accuracy) standards or standards prepared according to EPA Traceability Protocol #1. Adherence to prescribed QC procedures will ensure data of consistent and measurable quality. Analytical quality control will focus upon the use of control standards to provide a measure of analytical precision. Also, specific acceptance criteria are defined for various analytical operations, including calibrations, control standard analyses, drift checks, blanks, etc. The following general QC procedures will be incorporated into the analytical effort.

1. The on-site testing team leaders will review all analytical data and QC data on a daily basis for completeness and acceptability.
2. A master logbook will be maintained.
3. Analytical QC data will be tabulated using the appropriate charts and forms on a daily basis.
4. Copies of the QC data tabulation will be submitted to the QA Coordinator following each test, with all originals kept on file.
5. All hard copy raw data (i.e., chromatograms, computer printouts, etc.) will be maintained in organized files.

Specific analytical QC procedures for each of the instrumental analyses are discussed below.

7.2.1 CEMS Quality Control Procedures

During the testing program, CEMS operation will include the following general QC procedures.

1. CEMS data will be collected using a central computerized data acquisition system.
2. All calibration standards will be certified standards (±2% analytical accuracy).
3. All CEMS calibration data, QC data, and maintenance operations will be recorded in bound laboratory notebooks or on preformatted data sheets.

In addition to these general QC considerations, CEMS drift checks for CO, CO_2, O_2, NO_x, and THC will be performed daily to ensure that the monitoring data remain within precision and accuracy limits specified in the performance specifications. Drift will be defined as the difference between the monitor value and the actual concentration of the standard, expressed as a percentage of the span value. Drift checks will be performed prior to any calibration or zero adjustment. For the O_2 and CO_2 monitors, the acceptance limit is 0.5% O_2 or CO_2 (absolute). The acceptance limit for the CO, NO_x, and THC monitors is 5% drift.

Each day each monitor will be challenged with a mid-range (40-60%) certified gas. Reference to these gases will define accuracy and serve as a daily audit for each instrument. Table 5-7 specifies the audit standards as well as the calibartion standards.

7.2.2 Quality Control Procedures for GC/MS Analysis

Internal quality control checks on the GC/MS analysis will consist of daily tuning of the mass spectrometer, analysis of GC column performance standards, calibration checks, and analysis of surrogate spikes for extraction efficiency. A multipoint calibration curve and response factor for the compounds of interest will be developed.

7.2.2.1 Instrument Calibration and Tuning

Mass calibration of the GC/MS instrument will be demonstrated daily with perfluorotributylamine (PFTBA) prior to tuning the instrument to the criteria for bromofluorobenzene (BFB).

System Performance

System performance will be verified initially to ensure a minimum average response factor of 0.3 (0.25 for bromoform) for the following system performance check compounds (SPCCs):

- Chloromethane;
- 1,1-Dichloroethane;
- Bromoform;
- 1,1,2,2-Tetrachloroethane; and
- Chlorobenzene.

The above criteria will be demonstrated every 12 hours for the duration of the analyses.

Analyte Calibration

The initial five point calibration of 10, 20, 50, 100 and 200 ug/L standards, used for generating response factors, must demonstrate a percent relative standard deviation (%RSD) of less than 30 for all calibration check compounds (CCCs) shown below:

- 1,1-Dichloroethene;
- Chloroform;
- 1,2-Dichloropropene;
- Toluene;
- Ethylbenzene; and
- Vinyl chloride.

This criterion will also be demonstrated every 12 hours for the duration of the analyses for the 8240 analyses. VOST analyses using Method 5040 will employ an initial three point instrument calibration.

System Performance

System performance will be verified initially to ensure a minimum average response factor of 0.3 (0.25 for bromoform) for the following system performance check compounds (SPCCs):

- Chloromethane;
- 1,1-Dichloroethane;
- Bromoform;
- 1,1,2,2-Tetrachloroethane; and
- Chlorobenzene.

The above criteria will be demonstrated every 12 hours for the duration of the analyses.

Analyte Calibration

The initial five point calibration of 10, 20, 50, 100 and 200 ug/L standards, used for generating response factors, must demonstrate a percent relative standard deviation (%RSD) of less than 30 for all calibration check compounds (CCCs) shown below:

- 1,1-Dichloroethene;
- Chloroform;
- 1,2-Dichloropropene;
- Toluene;
- Ethylbenzene; and
- Vinyl chloride.

This criterion will also be demonstrated every 12 hours for the duration of the analyses for the 8240 analyses. VOST analyses using Method 5040 will employ an initial three point instrument calibration.

Surrogate Recovery

To monitor purging and trapping efficiency, each sample will be spiked with three surrogates before analysis. These compounds are listed below:

- 1,2-Dichloroethane-d_4;
- Toluene-d_8; and
- Bromofluorobenzene.

Blanks

Daily extraction blanks will be analyzed to check for background contamination. For measurement of the organic constituents collected by VOST at least one method blank and one field blank will be analyzed with each set.

Duplicate Matrix Spike Analyses

A sample from each matrix type will be used to perform duplicate matrix spike analyses. Duplicate aliquots will be spiked with 125 ng each of the following volatile compounds:

- 1,1-Dichloroethene;
- Trichloroethene;
- Chlorobenzene;
- Toluene; and
- Benzene.

Internal Standard

All volatile measurements will utilize the internal standard method of quantitation. The internal standards to be used are bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d5.

7.2.2.2 Semivolatiles

Instrument Tuning and Calibration

All semivolatile extracts will be analyzed using the procedures outlined in SW-846 Method 8270, third edition without significant modification. The mass spectrometer used for these analyses will be tuned daily using PFTBA. The stability of this tune will be verified by demonstrating an acceptable spectrum of decafluorotriphenylphosphene (DFTPP) daily (or every 12 hours). Chromatography will be verified by examining the peak shape of benzidine and pentachlorophenol daily.

System Performance

System performance will be verified daily, beginning with the 5-point calibration, by demonstrating that system performance check compounds (SPCCs) have response factors greater than 0.05 using the mid level calibration standard. These SPCCs are:

- N-Nitrosodi-n-propylamine;
- Hexachlorocyclopentadine;
- 2,4-Dichlorophenol; and
- 4-Nitrophenol.

Continuing Analyte Calibration

A 5-point calibrations will be generated before beginning this work using Appendix IX standards at 10, 20, 50, 100, and 200 ug/mL concentrations. Specific ion response factors for the calibration check compounds must have less than 30% relative standard deviation over the range calibrated. These calibration check compounds (CCCs) are:

- Phenol;
- 1,4-Dichlorobenzene;
- 2-Nitrophenol;
- 2,4-Dichlorophenol;
- Hexachlorobutadiene;
- 4-Chloro-3-methylphenol;
- Acenaphthene;
- 2,4,6-Trichlorophenol;
- N-nitrosodi-N-phenylamine;
- Pentachlorophenol;
- Fluoranthene;
- Di-n-octylphthalate; and
- Benzo(a)pyrene.

These CCCs will be reanalyzed every 12 hours to verify that the response factor remain with ±30% of that generated from the average of the 5 point standard.

Surrogate Recoveries

Surrogate recoveries for the semivolatile extraction surrogates will be determined for all blanks and samples.

Blanks

At least one extraction blank will be generated for each sample set. For Toxic Characteristic Leaching Procedures (TCLP) samples, a leachate and reagent blank will be generated and analyzed with the samples.

Duplicate Matrix Spike Analyses

A sample from each matrix type will be used to perform duplicate matrix spike analyses. Samples will be spiked prior to extraction with a mixture containing the following:

- 1,2,4-Trichlorobenzene;
- Acenaphthene;
- 2,4-Dinitrotoluene;
- Pyrene;
- 1,4-Dichlorobenzene;
- N-nitrosodi-n-propylamine;
- Pentachlorophenol;
- Phenol;
- 2-Chlorophenol;
- 4-Chloro-3-methyl phenol; and
- 4-Nitrophenol.

This analysis gives a measure of both accuracy and precision of the method.

Internal Standards

All semivolatile measurements will utilize the internal standard method of quantitation. The internal standards to be used are:

- 1,4-Dichlorobenzene-d4;
- Napthalene-d8;
- Acenaphthene-d10;
- Phenanthrene-d10;
- Chrysene-d12; and
- Perylene-d12.

7.2.2.3 Dioxins and Furans

Instrument Calibration and Tuning

PCDD/PCDFs will be analyzed according to the procedures outlined in SW-846 Method 8280, third edition. Mass calibration of the instrument will be demonstrated daily with PFTBA.

PCDD/PCDF Retention time Group Determination

Since selected-ion-monitoring mass spectrometry is used for PCDD/PCDF analysis, it is first necessary to determine the retention time windows for each isomer group. This is accomplished by injection of a PCDD/PCDF retention time standard which contains the first and last eluter from each isomer group. The isomer group retention time analysis is performed once at the beginning of each project and again if the retention times shift more than ±0.2 minutes. Adequate sensitivity is also verified from the retention time standard which contains 2,3,7,8-TCDD at a concentration of 0.2 ug/mL.

Chromatography Performance Check

A TCDD Chromatographic test mixture is analyzed daily to verify that there is at least 25% valley resolution between 2,3,7,8-TCDD and 1,2,3,4-TCDD.

PCDD/PCDF Calibration

Response factors for each PCDD/PCDF isomer class are determined from a five-point calibration at 0.1, 0.2, 0.5, 1.0, and 2.5 ug/mL concentrations. This five point calibration is performed in triplicate and the average response factors are calculated. The response factors determined for each isomer class must not have %RSD greater than 30%. Radian uses a calibration standard containing the following analytes:

- 2,3,7,8-TCDD;
- 1,2,3,7,8-PeCDF;
- 2,3,4,7,8-PeCDF;
- 1,2,3,8,9-PeCDF;
- 1,2,3,4,7,8-HxCDF;
- 1,2,3,4,8,9-HxCDF;
- 1,2,3,4,6,7,8-HpCDF;
- OCDF;

- 2,3,7,8-TCDF;
- 1,2,3,7,8-TCDF;
- 1,2,3,7,8-PeCDD;
- 1,2,3,4,7-PeCDD;
- 1,2,3,4,7,8-HxCDD;
- 1,2,3,4,6,7,8-HpCDD; and
- OCDD.

Where more than one isomer per isomer group is present, the average response factor is used.

For quantitation, the following mixture of internal standards is contained in the calibration standard solutions. This mixture is also added to each sample before extraction:

- $^{13}C_{12}$ - 2,3,7,8-TCDD;
- $^{13}C_{12}$ - 2,3,7,8-TCDF;
- $^{13}C_{12}$ - 1,2,3,7,8-PeCDD;
- $^{13}C_{12}$ - 1,2,3,7,8-PeCDF;
- $^{13}C_{12}$ - 1,2,3,6,7,8-HxCDD;
- $^{13}C_{12}$ - 1,2,3,4,7,8-HxCDF;
- $^{13}C_{12}$ - 1,2,3,4,6,7,8-HpCDD;
- $^{13}C_{12}$ - 1,2,3,4,6,7,8-HpCDF;
- $^{13}C_{12}$ - OCDD; and
- $^{13}C_{12}$ - OCDF.

Each isomer class is quantitated using a C-13 internal standard from that class.

Daily Response Factor Check

The 1.0 ug/mL PCDD/PCDF standard is analyzed at the beginning of each working day to verify that the response factors obtained for that day agree to within ±30% of those obtained from the initial five-point calibration in triplicate. Also, isotope ratios for each PCDD/PCDF isomer class are verified to be within ±15% of their theoretical values.

Surrogate Recovery Determinations

The mixture of internal standards added to each sample before extraction also serves as the surrogate solution. Surrogate recoveries are determined relative to $^{13}C_{12}$ - 1,2,3,4-TCDD which is added to each extract prior to analysis. Surrogate response factors are determined from analysis of a $^{13}C_{12}$ - 1,2,3,4-TCDD spiked aliquot of the internal standard/surrogate solution.

Blank Analysis

A laboratory extraction blank is generated for each batch of samples or every 20 samples, whichever is less. These blanks will go through all the specified extraction and cleanup steps. Surrogate recoveries and background contamination levels (if any) are determined for each blank.

QC Check Sample

An EPA or NBS check sample is analyzed after every five-point calibration to determine that the PCDD/PCDF standards and calibration curve used are acceptable.

7.2.3 Metals Analyses

Instrument Calibration

All samples will be analyzed according to the procedures outlined in the appropriate methods listed in Section 5. Calibration of all instrumentation will be performed daily with freshly prepared standards.

System Performance

System performance will be verified immediately after calibration with a calibration verification standard prepared from a different source from the calibration standards. An NBS or EPA Quality Assurance sample will be used for this purpose when applicable. This standard is then analyzed at 10% intervals throughout the analysis and at the end of the analytical run, to verify system control.

Blanks

Calibration of DI water blanks will be analyzed at a frequency of 10% to check for system contamination. Preparation blanks, aliquots of deionized water that are taken through the digestion/extraction process as though they were a sample, will be prepared at a frequency of one per batch digested/extracted or one per twenty samples whichever is greater.

ICP Interference Check Sample

An ICP Interference Check Sample will be analyzed at the beginning, middle and end of the run throughout most of the project. The results must be within the 80-120% recovery control limits set by the EPA.

Duplicate Matrix Spike Analysis

A duplicate matrix spike sample will be analyzed once during the project. The relative percent difference should be within 20 percent.

7.2.4 Total Organic Carbon

Instrument Calibration

All samples will be analyzed according to the procedures outlined in EPA Method 415.1. Initial calibration will be performed daily with freshly prepared standards.

Calibration will be verified immediately after initial calibration with an independently prepared control sample. The standard is then analyzed every 10 samples to verify system control.

Blanks

A reagent blank will be analyzed every 20 samples to indicate system contamination.

Duplicate Analyses

A duplicate sample will be analyzed every 20 samples. The relative percent difference (RPD) should be within 20 percent.

7.2.5 Quality Control Procedures for Chloride Analyses

Following Method 27 from the "FGD Chemistry and Analytical Methods Handbook", Radian Corporation, Volume 2, July 1984, a Dionex Ion Chromatograph will be used to determine chloride concentrations in the impinger catch from the HCl train. This analysis method involves the generation of a standard calibration curve. This curve is a linear plot of analyte concentration versus instrument response (conductivity or absorbance). The calibration curve is generated using response data from analysis of the blanks and calibration standards. This data is plotted mathematically using linear regression to get the slope, y- intercept, and correlation coefficient of the calibration curve. The validity of the resulting curve may be assessed by examining these three items. The slope of the curve is related to response sensitivity. Any marked deviation from the average slope for the method and/or parameter of interest indicates that a change in sensitivity has occurred. The acceptance criterion for slope will be agreement with running mean slope for six most recent determinations within 10%. The y-intercept of the curve should ideally be equal to zero, i.e., instrument response to a blank should be zero and the calibration curve should pass through the origin. In practice, the curve rarely passes exactly through the origin.

The correlation coefficient is indicative of the linearity of the curve. Perfect correlation of the x and y data points is indicated by a correlation coefficient of 1.0. An acceptable calibration curve will have a correlation coefficient $\geq$0.9950.

7.2.6 Quality Control for Fixed Gas Analysis

Fixed gas analysis for molecular weight determination will be based on EPA Method 3. Quality control procedures will include two or more single point calibrations prior to the field test activities. The acceptance criteria for the single point calibrations will be duplicate analysis within $\leq$5% (CV). A control standard will also be analyzed prior to the test to provide an estimate of accuracy. The acceptance criteria for the control standard will be agreement within 10% of the theoretical value.

8.0 PERFORMANCE AND SYSTEM AUDITS

No performance or systems audits are planned as an integral aspect of this program to be performed by Radian. Radian will cooperate with the EPA or other regulatory agencies with Agency-requested performance and/or systems audits. Any performance and/or systems audits requested will be coordinated through Mr. M. J. Krall of Radian Corporation.

9.0 DATA QUALITY INDICATORS

The QC analyses conducted during the testing program are designed to provide a quantitative assessment of the measurement system data. The two aspects of data quality which are of primary concern are precision and accuracy. Accuracy reflects the degree to which the measured value represents the actual or "true" value for a given parameter and includes elements of both bias and precision. Precision is a measure of the variability associated with the measurement system. The completeness of the data will be evaluated based upon the valid data percentage of the total tests conducted.

9.1 Precision

Precision by the definition presented in the EPA Quality Assurance Handbook for Air Pollution Measurement Systems. Volume I: Principles (EPA-600/976-005) is "a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions." Different measures of precision exist, depending upon these "prescribed similar conditions."

Quality control procedures, such as control sample analyses and replicate analyses, represent the primary mechanism for evaluating measurement data variability or precision. Replicate analyses will be used to define analytical replicability, while results for replicate samples may be used to define the total variability (replicability) of the sampling/analytical system as a whole.

Precision of the measurement data for this program will be based upon replicate analyses (replicability) and control sample analyses (repeatability). Where there are only two data points (e.g., replicate or repeat analysis), variability will be expressed in terms of the relative percent difference (RPD). The RPD is defined as:

$$\% \text{ RPD} = \frac{\text{Maximum Value} - \text{Minimum Value}}{[\,(\text{Maximum Value} + \text{Minimum Value})/2]} \times 100,$$

and is commonly used to express variability between duplicate measurements because it is easily calculated and measurement distribution is always normal for duplicate results. For data sets greater than two points, the coefficient of variation (CV) is more commonly used. It is defined by the equation

$$\% \text{ CV} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100.$$

Both terms are independent of the error (accuracy) of the analyses and reflect only the degree to which the measurements agree with one another, not the degree to which they agree with the "true" value for the parameter measured. To facilitate comparison of variability for data sets of two and more, the two quantitaties for two data points are related by

$$\% \text{ RPD} = \sqrt{2} \times (\% \text{ CV})$$

9.2 Accuracy

Accuracy is the agreement between a measurement value and a theoretical value. Accuracy will be expressed as the relative error (% RE) where:

$$\% \text{ RE} = \frac{\text{Measured Value} - \text{Theoretical Value}}{\text{Theoretical Value}} \times 100,$$

This accuracy estimate includes error due to both imprecision and bias.

The accuracy objectives in Table 5-3 represent error for analysis of target analytes spiked in the actual sample matrix, except for continuous flue gas measurements. Accuracy estimates for CEMs will be based on the critieria presented in Section 5.19.7.

The analytical systems will also be checked routinely for system accuracy in the absence of matrix effects. The following equation will be used to calculate matrix spike recovery:

$$\%\ \text{Recovery (spike)} = \frac{C_{ss} - C_{us}}{C_{sa}} \times 100$$

where:

C_{ss} = Analyte concentration in spiked sample;
C_{us} = Analyte concentration in unspiked sample; and
C_{sa} = Analyte concentration added to the sample.

If matrix spike error exceeds the specified acceptance criteria, a QC check sample (analyte spiked in DI water) will be analyzed to determine whether the error was due to matrix interferences or to an assignable cause of error in the analytical system itself. Data which are not within the acceptance criteria for matrix spikes will be flagged.

Daily control sample analyses represent one mechanism for controlling measurement system error. Typically, repeated measurements are made of the parameter of interest for the same control sample or for additional samples at different levels, and the average error is calculated. This error value represents an estimate of measurement bias or systematic error, although it is often simply referred to as "accuracy." The significance of the bias estimate may be evaluated using confidence intervals. An approximate 95% confidence interval for the mean error (bias) can be calculated using:

$$\text{Mean (X)} \pm t_{.025,\ (n-1)} \frac{\text{Standard Deviation}}{\sqrt{n}}$$

where n is the number of measurements used to compute the average and standard deviation and $\pm$ t .025, (n-1) is a table statistical value (0.025 confidence level, n - 1 degrees of freedom; when n is greater than 10, t approaches 2.0).

As an example, for a particular set of nine measurements, assume an overall mean of 20 ppm is reported, and the standard deviation of these data is 10 ppm. Also, assume that the true concentration is 30 ppm. For these measurements, the 95% confidence interval is:

$$20 \pm 2.3 \frac{10}{\sqrt{9}} \qquad \text{or} \qquad 20 \pm 7.7$$

which is the interval (12,28). Since this interval does not include the true value, 30 ppm, a conclusion of bias is justified. The magnitude of this bias is between 2 and 18 ppm. The uncertainty in the estimate is due to variability arising from random error.

Daily control samples analyses will be used to assess measurement bias. The average degree of agreement between measured values and actual values for control samples provides a long-term, or average estimate of measurement bias, as well as precision (repeatability).

9.3 Completeness

Measurement data completeness is a measure of the extent to which the data base resulting from a measurement effort fulfills objectives for the amount of data required. For this program, completeness will be defined as the valid data percentage of the total tests planned. As previously shown in Table 3-1, this valid data percentage will be 90% for all tests.

9.4 Method Detection Limits

Method detection limits are determined by multiplying instrument detection limits by a factor based on the initial sample volume or mass and the final extraction volume used for analysis. For organic analyses, one liter extraction volumes are generally used for aqueous samples, ten grams for solids. For flue gas derived samples, the entire XAD, filter and rinse samples are co-extracted. The amount of liquid or solid sample used is matrix dependent; however, it will generally be less for samples with higher concentrations of extractable materials.

The instrumental detection limits (in ug/L for ICP analyses) shall be determined by multiplying by 3, the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration 3-5 times the IDL on three (3) nonconsecutive days with 7 consecutive measurements per day.

For GC/MS analyses, the detection limit is determined by a 5:1 signal to noise ratio for the primary confirmation ion (fragment) and requires the presence or detection of the secondary confirmation ion.

10.0 CORRECTIVE ACTION

During the course of the Shirco Portable Pilot Test Unit demonstration program, it will be the responsibility of the field task leader and the sampling team members to see that all measurement procedures are followed as specified and that measurement data meet the prescribed acceptance criteria. In the event a problem arises, it is imperative that prompt action be taken to correct the problem(s). The field team leaders will initiate corrective action in the event of QC results which exceed acceptability limits. Corrective action may also be initiated by the team leaders upon identification of some other problem or potential problem. Corrective action may be initiated by the QA Coordinator based upon QC data or audit results. The corrective action scheme is shown in the form of a flow chart in Figure 10-1.

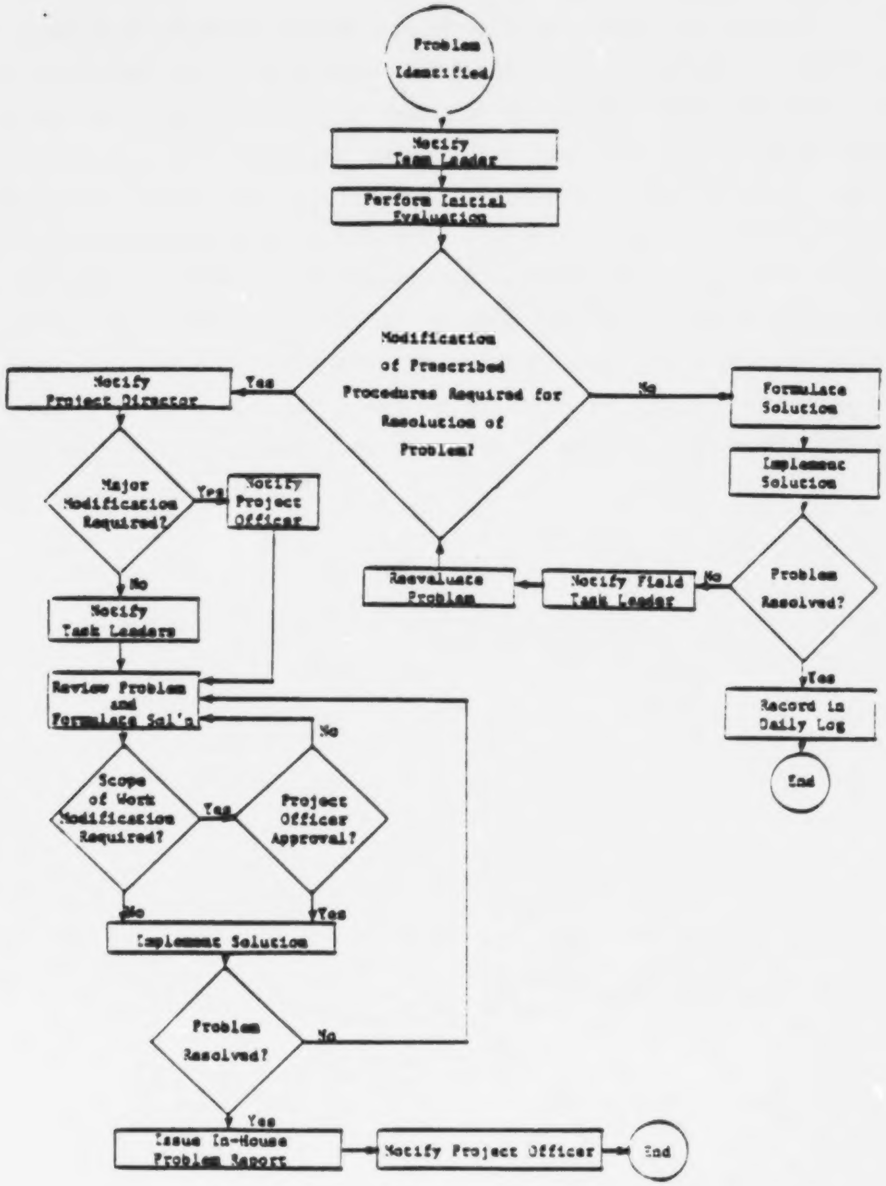

Problem Identified
Notify Task Leader
Perform Initial Evaluation
Modification of Prescribed Procedures Required for Resolution of Problem?
Yes
No
Notify Project Director
Major Modification Required?
Yes
Notify Project Officer
No
Notify Task Leaders
Review Problem and Formulate Sol'n
Scope of Work Modification Required?
Yes
Project Officer Approval?
No
No
Yes
Implement Solution
Problem Resolved?
No
Yes
Issue In-House Problem Report
Notify Project Officer
End
Formulate Solution
Implement Solution
Problem Resolved?
No
Notify Field Task Leader
Reevaluate Problem
Yes
Record in Daily Log
End

Figure 10-1. Corrective Action Flow Scheme

11.0 QUALITY ASSURANCE REPORTING

Effective management of a field sampling and analytical effort requires timely assessment and review of field activities. This will require effective interaction and feedback between the field team leader, the Project Director, and the QA Coordinator.

During the Shirco incinerator test, the field team leader will submit a verbal QC report to the Radian QC Coordinator. This report will address the following:

- summary of activities and general program status;
- summary of corrective action activities;
- assessment and summary of data completeness; and
- summary of any significant QA/QC problems and recommended and/or implemented solutions not included above.

The project final report will include a separate QA/QC section which addresses QA/QC aspects of the project.

Problems requiring swift resolution will be brought to the immediate attention of the Project Director via the corrective action scheme discussed in Section 10.

12.0 PROJECT SAFETY

A primary objective of the testing activity is that it be performed in a safe and prudent manner. The site is a controlled, limited-access facility that presently meets or exceeds applicable safety standards. Radian will comply strictly with the facility's safety program. However, in order to minimize any risks, the Radian team will be equipped and prepared for emergency situations likely to occur.

The following discussion contains information regarding general safety procedures and site-specific safety considerations.

12.1 General Safety Procedures

The safety procedures and protective equipment required on site will vary according to the type of activity in progress and one's proximity to the sampling area. The field task leader will serve as safety coordinator and will decide the types of protective equipment to be used and the delineation of safety zones on the job site.

12.1.1 Safety Zones

Safety zones will be established according to the exposure levels encountered. It is anticipated that only Level-One and Level-Two zones will be required at this work site. The following three-level exposure system will be used:

- Level One--The exposure level is very low hazard associated with site activities. Low hazard area requires little or no safety equipment.
- Level Two--An exposure level exists which requires intermediate precautions. Hazard potential is defined and safety equipment is required.

- Level Three--An extreme exposure level exists and strict safety precautions are required. This exposure level is considered life-threatening.

No Level Three exposure is considered likely to occur. The safety coordinator will inform workers as to what safety zone they will be in and what safety equipment will be required prior to field testing.

12.2 Safety Equipment

Equipment will be available to protect each worker from Level Two exposure. In addition, the following equipment will be available on site:

- Ear protection;
- Emergency eye washes; and
- First aid supplies.

12.3 Safety Training

Prior to the initiation of on-site work, the field task leader will conduct a safety meeting. The meeting will inform the project personnel of the potential and real hazards present, instill in them the necessity for safety precautions, and develop their understanding of proper safety measures. Instructions as to the use of various safety equipment and procedures will be given if necessary. Also, a contingency plan for emergency situations will be discussed.

12.4 Site-Specific Safety Procedures

Radian personnel will comply strictly with all the safety requirements of the facility, particularly with respect to hard hats, steel-toed boots, eye protection, and long-sleeved shirts.

Chemical exposure may be minimized by the use of protective clothing, respirators equipped with acid/organic canisters, disposable gloves, and Tyvek© suits when working in the immediate vicinity of the high level sources. The necessity of these measures will be decided by the project safety officer. Gloves will be worn when handling solid and liquid samples and filling sampling containers.

APPENDIX A

27.

Analysis of Anions (Fluoride, Chloride, Sulfite, Sulfate, and Phosphate) in Scrubber Liquors and Solids by Ion Chromatography with Modified Anion Eluent

1.0 DESCRIPTION OF METHOD

This procedure uses ion chromatography to analyze fluoride, chloride, sulfate, sulfite, and phosphate in scrubber liquors and total sulfur as sulfate in scrubber solids. The ions are separated on anion exchange resins and detected conductimetrically. The five anions of interest are separated with a Dionex fast-run AS3 separator. An AG3 guard column precedes the separator column. A packed-bed suppressor column is between the separator and the conductivity detector. The standard anion eluent is modified in this procedure to improve peak resolution. Formaldehyde is used as a sample preservative to inhibit sulfite oxidation and to allow direct sulfite and sulfate analysis in the same sample.

Eluent and injected solution first enter the guard column where most particulates and organic contaminants are trapped before they can reach and possibly damage the separator. Separation occurs according to anion size and resin affinity in the next column, the anion separator. Eluent and separated anionic species then enter the suppressor column where response to the background eluent ions is eliminated. The conductivity cell detects the separated anions and produces an electronic response that is recorded with a strip chart recorder as a function of time. For a given set of conditions, the elution time is characteristic of the species measured. Peak heights are proportional to species concentrations. The peak heights recorded for sample injections are compared with calibration curves for each anion to determine concentration in the sample.

2.0 SIGNIFICANCE AND USE

This procedure can be used to analyze a number of different anions in both liquid and solid samples from FGD systems provided high molar ratios of species do not prevent resolution of the peaks of interest. This method has two significant advantages over other methods for the anions of interest. First, it is faster than analyzing the anions separately using a different method for each anion. Second, it allows the direct determination of sulfite and sulfate in one sample. The other alternatives for sulfite and sulfate analysis require immediate determination of sulfite on one sample of a material at the time of sampling, oxidation of sulfite to sulfate in another sample, determination of total sulfate on the oxidized sample, and calculation of sulfate by difference.

This method can be used to determine chloride and sulfate in samples from lime, limestone, dual alkali and magnesia FGD systems. It can be used for chloride, sulfite, and sulfate in all but high TDS systems in which sulfite concentration is very low (8-10 mg/L). Its application for fluoride and phosphate analysis is probably limited to low TDS lime or limestone systems in which chloride and sulfate concentrations are low enough to allow peak resolution. There are little or no reliable data to indicate typical levels of fluoride and phosphate in scrubber liquors, so it is possible that the actual concentrations of these anions are too low to be measured by this method.

3.0 DETECTION LIMIT AND RANGE OF METHOD

Maximization of instrument sensitivity was not an objective in developing this procedure. The detection limit and range for the operating conditions used in this procedure (30 µmhos/cm, 50% modified eluent, and 33% flow) are shown in Table 1.

Calibration lines based on peak heights (method recommended rather than peak area by the instrument manufacturer) are nonlinear for peak heights less than about 10 percent of full scale. The lowest standard should have a concentration so that the peak height is about 20 percent of full scale. The detection limits listed in Table 1 have been defined as one-half the concentration of the lowest standard, and the preferred range for measurement as the range from the concentration of the lowest standard to the concentration of the highest standard. Concentrations above the upper limit can be brought into the preferred range by dilution.

Table 1

ANION DETECTION LIMITS AND MEASUREMENT RANGES

Anion	Detection Limit (mg/L)	Range (mg/L)	
		Low	High
F^-	0.247	0.494	3.95
Cl^-	0.709	1.42	8.51
SO_3^{-2}	2.40	3.20	22.4
SO_4^{-2}	1.92	3.84	26.9
PO_4^{-3}	3.80	7.60	22.8

4.0 ELAPSED TIME AND LABOR HOURS REQUIRED

Reagent preparation requires between 90 and 120 minutes before initial instrument operation. Other daily preparations average between 15 and 30 minutes.

Assuming that a stable baseline has been established and that all standard solutions have been prepared, analysis of the first sample within the concentration range of the standards takes approximately 78 minutes, 35 of which involve operator time. This allows time for injection of a blank, four standards for a calibration curve, and a sample. Each additional sample will take 13 minutes to analyze. Instrument start-up and baseline stabilization will add 40 minutes to the time required from receipt of sample to final answer. Since calibration should be repeated within 4 hours of start-up, a total of 10 to 13 samples may be analyzed per calibration. The actual number of samples analyzed depends on anion concentrations in the samples, which affect elution time and suppressor column capacity. If sample concentrations in original dilutions are not in the preferred range, additional dilutions and injections must be made. This reduces the number of samples that can be analyzed per calibration. An ASC-2 packed-bed suppressor column can normally be operated 6 to 8 hours before regeneration is necessary. A steady upward drift of the baseline indicates the need for regeneration.

5.0 DEFINITIONS

anion exchange resin - a synthetic organic exchange material capable of the reversible exchange of negatively charged ions

regeneration - the part of the operating cycle of an ion-exchange process in which a chemical solution is passed through the ion-exchange bed to prepare it for service runs

retention time - the time that elapses from the introduction of the sample until the component peak maximum is reached

eluent - liquid medium that carries injected samples/solutions through the ion separator column

conductivity cell - an analytical detector that measures electrical conductivity

6.0 INTERFERENCES/SOURCES OF ERROR

The main source of interference in ion chromatography is overlapping or unresolved peaks. To help reduce peak overlap and to keep analysis time as short as possible, a 50% strength modified anion eluent (0.0013 M NaOH and 0.00175 M Na_2CO_3) at a flow rate of 33% is used. Retention times at these conditions are given in Table 2. Interference considerations are noted below.

Table 2

AVERAGE RETENTION TIMES USING 50 PERCENT MODIFIED ELUENT AT A FLOW RATE OF 33 PERCENT

Species	Retention Time (minutes)
Fluoride	2.2
Chloride	2.9
Sulfite	5.9
Sulfate	7.8
Phosphate	11.2

Phosphate peak resolution is not achievable at these conditions in solutions containing high sulfate-to-phosphate molar ratios. Peak resolution might be achieved at other conditions, but the analysis time per sample might be unacceptably long.

Sulfate-to-sulfite molar ratios of 75:1 or greater prevent adequate separation of the sulfite peak in solutions with low sulfite concentration.

Fluoride peak resolution is affected by chloride present in molar concentrations greater than sixty times the fluoride concentration and sulfate present in molar concentrations greater than 200 times the fluoride concentration.

This procedure calls for addition of formaldehyde as a sample preservative at the time of sample collection. Formaldehyde is added at 5 times the sulfite concentration to retard or inhibit sulfite oxidation. Addition of formaldehyde at 5 times the sulfite concentration to sulfite, sulfate, chloride, fluoride, and phosphate standards showed that formaldehyde had no effect on analysis of standards and no formaldehyde interference is expected in analysis of any of the anions in scrubber liquors.

7.0 ALTERNATE METHODS

Ion chromatography provides rapid analyses of anions in scrubber samples. The cost of purchasing the instrument and training operators often must be justified by the number of samples to be analyzed. Several methods for analyzing each of the anions covered in this procedure are provided in this handbook. The choice of method will depend on the concentration range of the anion in the sample, the time required to perform the procedure, interferences, the degree of accuracy required, and the number of samples to be analyzed. These elements are summarized in Tables 3 through 7 for the chloride, fluoride, sulfate, sulfite and phosphate methods included in this handbook.

8.0 PRECAUTIONS/CRITICAL STEPS

- Columns may be damaged if not thoroughly rinsed with deionized water between eluent and regenerant cycles.
- Greater resolution between peaks may be achieved by:
  - Use of a weaker eluent,
  - Addition of separator columns (the greater the number of active separator sites, the greater the possible resolution),
  - Decrease in flow rate.
- Never increase flow rate when columns are in line, or when pump is not operating. Doing so may result in liquid leaks at column fittings or damage to the pump's internal flow control mechanism.
- For instrument problems consult the ion chromatograph operators' manual.
- Different dilutions (and therefore separate injections) may be required for determination of species present in high and low concentrations. The conditions specified in this procedure will not yield adequate resolution of peaks if the molar ratio of two species with similar retention times is very large. Section 6 specifies the molar ratios of concern for this procedure.

Table 3

CHLORIDE METHODS COMPARISON

Method	Range	Interferences/ Sources of Bias	Applications	Preparation Time[a] (hr)	Sample Analysis Time[b] (hr/single sample)	Single Operator Coefficient of Variation[c] (%)	Special Equipment Requirements
Ion Chromatography—50% Modified Anion Eluent	1.4 to 8.5 mg/L in the aliquot injected	Overlapping peaks, which may be resolved by changing eluent strength or flow rate	Liquors from lime, limestone, dual alkali, and magnesia processes	3.25	0.25	12.3	Ion chromatograph with anion separator and suppressor columns Strip chart recorder
Ion Chromatography—Standard Anion Eluent	1.4 to 8.5 mg/L in the aliquot injected	None	Liquors from lime, limestone, dual alkali and magnesia processes	2.75	0.15	1.7	Ion chromatograph with anion separator and suppressor columns Strip chart recorder
Mercuric Nitrate Titration	0.5 to 250 mg/L	Sulfite >10 mg/L (oxidize with peroxide) Iron III >10 mg/L (reduce with hydroquinone) Chromate >100 mg/L (reduce, use modified indicator)	Liquors from lime, limestone, dual alkali and magnesia processes. Applicability depends on presence of interfering ions.	2.5	0.35	3.0	Microburet
Silver Nitrate Titration with Chromate Indicator	2.5 to 100 mg/L	Iron II >10 mg/L Orthophosphate at 25-250 mg/L (uncommon except when cooling water blowdown is used for make-up water) pH (adjust to neutral or slightly alkaline) Turbidity (add $Al(OH)_3$ and filter) Sulfite and thiosulfate (oxidize with peroxide) Bromide, iodide, cyanide and sulfide (uncommon in scrubber liquors) Magnesium and sulfate at 200 times the chloride molar concentration	Liquors from lime, limestone, dual alkali and magnesia processes. Applicability depends on presence of interfering ions.	2.5	0.35	2.3	Microburet

Table 3 (continued)

CHLORIDE METHODS COMPARISON

Method	Range	Interferences/ Sources of Bias	Applications	Preparation Time[a] (hr)	Sample Analysis Time[b] (hr/single sample)	Single Operator Coefficient of Variation[c] (%)		Special Equipment Requirements
Potentiometric Titration with Silver Nitrate	5.0 to 100 mg/L	Chromate and dichromate (reduce to chromic ion) Ferric iron at concentrations substantially higher than chloride concentration Bromide, iodide, ferricyanide (uncommon in scrubber solutions) Sulfite and thiosulfate (oxidize with peroxide)	Liquors from lime, limestone, magnesia, and dual alkali systems	2.0	0.20	Pure liquors Liquors containing interferences	2.5 5.0	pH meter with millivolt scale, electrodes, microburet

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[a] Includes labor hours for equipment set up, instrument calibration, reagent preparation and standardization

[b] Elapsed time from receipt of one sample to calculated final result including sample preparation, analysis, and calculations. Clean-up time is not included. Actual labor hours are less than elapsed time in some cases. Labor hours per sample will be reduced if more than one sample is analyzed.

[c] Coefficient of Variation (CV) = (standard deviation/mean) x 100

$$CV = \frac{[\Sigma(X_i - \bar{X})^2/n - 1]^{1/2}}{\bar{X}} \times 100$$

where: X_i = individual measurement

n = number of replicates

$\bar{X}$ = mean of measurements

Table 4

FLUORIDE METHODS COMPARISON

Method	Range	Interferences/ Sources of Bias	Applications	Preparation Time[a] (hr)	Sample Analysis Time[b] (hr/ single sample)	Single Operator Coefficient of Variation[c] (%)	Special Equipment Requirements
Ion Chromatography with Modified Eluent	0.5 to 4 mg/L in aliquot injected	Chloride >60 times fluoride concentration interferes. Sulfate >200 times fluoride concentration interferes.	Liquors from low TDS lime or limestone FGD systems	Reagent and instrument prep-2.2 Calibration-1.0	0.25	14.9 (est.)	Ion chromatograph with anion separator and suppressor columns Strip chart recorder
SPADNS Colorimetric	0.10 to 2.5 mg/L	Chloride (add Ag_2SO_4 to distillation flask.) Sulfate, chloride, iron, aluminum, alkalinity, phosphate (acidify sample and distill.)	Unverified	3.5	Liquid 0.40 Solid 1.15 Add 1 hr for sample distillation.	Single operator 27 (est.) Multilab 16	Distillation glassware Spectrophotometer with cuvettes
Ion Selective Electrode	0.20 to 1000 mg/L	Polyvalent cations Al (III), Fe (III), Si (IV), (add pH 5 buffer containing chelating agent.) Fluoroborates (Distill the sample.)	Unverified	3.5	Liquid 0.40 Solid 1.15 Add 1 hr for sample distillation.	Single operator 2.4 (est.) Multilab 4.8	pH/millivolt meter Fluoride and reference electrodes

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[a] Includes labor hours for equipment set up, instrument calibration, reagent preparation and standardization

[b] Elapsed time from receipt of one sample to calculated final result including sample preparation, analysis, and calculations. Clean-up time is not included. Actual labor hours are less than elapsed time in some cases. Labor hours per sample will be reduced if more than one sample is analyzed.

[c] Coefficient of variation (CV) = (standard deviation/mean) x 100

$$CV = \frac{\left[\sum (X_i - \bar{X})^2/n-1\right]^{1/2}}{\bar{X}} \times 100$$

where: X_i = individual measurement

n = number of replicates

$\bar{X}$ = mean of measurements

Table 5

SULFATE/TOTAL SULFUR METHODS COMPARISON

Method	Range	Interferences/ Sources of Bias	Applications	Preparation Time^a (hr)	Sample Analysis Time^b (hr/single sample)	Single Operator Coefficient of Variation^c (%)	Special Equipment Requirements
Gravimetric	50 to 250 mg/L	Sulfite (oxidize with peroxide) Silica (uncommon at interfering levels) Suspended matter (remove by filtration)	Liquors and solids from all FGD systems	3.0	2.6 to 21 hrs, depending on length of digestion	Liquors 4.3 Solids 8.3	
Turbidimetric	5 to 40 mg/L	Sulfite (oxidize with peroxide) Silica (uncommon in FGD systems at interfering levels) Suspended matter (remove by filtration) Highly colored liquors (analyze samples without BaCl₂ addition)	Liquors and solids from all FGD systems	4.5	Liquid 0.40 Solid 0.50	Liquors 5.7 Solids 6.6	Spectrophotometer or nephelometer, cuvettes
Barium Chloride/ Thorin Titration	5 to 1000 mg/L	Potassium, iron, chromium, aluminum (pass sample through cation exchange column) Sulfite (oxidize with peroxide) Sulfide (uncommon in scrubber systems) Nitrate, phosphate, and fluoride (uncommon at levels high enough to interfere) Chloride concentrations greater than sulfate concentration obscure titration end point	Most FGD systems (not applicable to solutions with chloride concentrations much greater than sulfate concentration)	3.0	Liquid 0.50 Solid 0.60	Liquors 1.23 Solids 3.5	Burets Ion exchange column
Acid-Base Titration	96 to 2880 mg/L	Phosphate is also determined by this method, but is not usually present in significant amounts Carbonate/carbon dioxide (stop titration at pH 4.6) Sulfite (oxidize with peroxide) Resin particles in effluent (filter)	Liquors from lime/ limestone, magnesia, and dual alkali processes	3.0	3.0 to 7.0	1 (est.)	Microburets Ion exchange columns

Table 5 (continued)

SULFATE/TOTAL SULFUR METHODS COMPARISON

Method	Range	Interferences/ Sources of Bias	Applications	Preparation Time[a] (hr)	Sample Analysis Time[b] (hr/single sample)	Single Operator Coefficient of Variation[c] (%)	Special Equipment Requirements
Ion Chromatography--50% Modified ion Eluent	3.8 to 27 mg/L in the aliquot injected	None	Liquors and solids from lime, limestone, dual alkali, and magnesia FGD processes	3.25	0.25	Liquors 3.7 Solids 4.1	Ion chromatograph with anion separator and suppressor columns Strip chart recorder
Ion Chromatography--Standard Anion Eluent	3.8 to 15 mg/L in the aliquot injected	None	Liquors and solids from lime, limestone, dual alkali, and magnesia FGD processes	2.75	0.15	0.75	Ion chromatograph with anion separator and suppressor columns Strip chart recorder

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[a] Includes labor hours for equipment set up, instrument calibration, reagent preparation and standardization

[b] Elapsed time from receipt of one sample to calculated final result including sample preparation, analysis, and calculations. Clean-up time is not included. Actual labor hours are less than elapsed time in some cases. Labor hours per sample will be reduced if more than one sample is analyzed.

[c] Coefficient of variation (CV) = (standard deviation/mean) x 100

$$CV = \frac{[\Sigma(X_i - \bar{X})^2/n - 1]^{1/2}}{\bar{X}} \times 100$$

where: X_i = individual measurement
n = number of replicates
$\bar{X}$ = mean of measurements

Table 6

SULFITE METHODS COMPARISON

Method	Range	Interferences/ Sources of Bias	Applications	Preparation Time[a] (hr)	Sample Analysis Time[b] (hr/single sample)	Single Operator Coefficient of Variation[c] (%)	Special Equipment Requirements
Ion Chromatography—50% Modified Anion Eluent	3.2 to 22.4 mg/L in aliquot injected	Overlapping or unresolved peaks (change eluent strength or flow rate) Sulfate to sulfite molar ratios ≥ 75 to 1	Liquors from all but high TDS systems in which sulfite concentration is very low (8-10 mg/L)	3.25	0.25	Liquids 80-40,000 mg/L 2.5	Ion chromatograph with anion separator and suppressor columns, strip chart recorder
Direct Iodine Titration	0.8 to 248 mg/L	Copper II and ferrous iron (complex with EDTA) Nitrite (destroy by sulfonic acid addition) Sulfide, thiosulfate, organic matter	Liquors from lime, limestone, magnesia, and dual alkali systems Not applicable to solids—cloudy solution masks starch end point	2.5	0.20	6.4	Buret
Iodine-Thiosulfate Titration	8.0 to 800 mg/L	Air oxidation of iodine (follow storage precautions) Extremes of pH (buffer samples to pH 6) Sulfide, thiosulfate, tin, iron, lead (not typically found in scrubber systems)	Liquors and solids from lime, limestone, dual alkali, magnesia systems	2.6	Liquids 0.25 Solids 0.50	Liquids 80-151 mg/L 4.6 2300-39000 mg/L 1.2 Solids Lime scrubber 0.9 Lime spray dryer 6.6	Buret

Table 6 (Continued)

SULFITE METHODS COMPARISON

Method	Range	Interferences/ Sources of Bias	Applications	Preparation Time[a] (hr)	Sample Analysis Time[b] (hr/single sample)	Single Operator Coefficient of Variation[c] (%)		Special Equipment Requirements
Iodine-Phenylarsine Oxide Titration	8.0 to 800 mg/L	Air oxidation of iodine (follow storage precautions) Extremes of pH (buffer samples to pH 6) Sulfide, thiosulfate, tin, iron, lead (not typically found in scrubber systems)	Liquors and solids from lime, limestone, dual alkali, magnesia systems	2.6	Liquids 0.25 Solids 0.50	Liquids 88-151 mg/L 2300-39000 mg/L Solids	 3.5 0.92 1.4	Buret
Iodine-Arsenite Titration	8.0 to 800 mg/L	Air oxidation of iodine (follow storage precautions) Extremes of pH (buffer samples to pH 6) Sulfide, thiosulfate, tin, iron, lead (not typically found in scrubber systems)	Liquors and solids from lime, limestone, dual alkali, magnesia systems	2.6	Liquids 0.25 Solids 0.50	Liquids 88-151 mg/L 2300-39000 mg/L Solids	 5.6 1.5 1.1	Buret

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[a] Includes labor hours for equipment set up, instrument calibration, reagent preparation and standardization

[b] Elapsed time from receipt of one sample to calculated final result including sample preparation, analysis, and calculations. Clean-up time is not included. Actual labor hours are less than elapsed time in some cases. Labor hours per sample will be reduced if more than one sample is analyzed.

[c] Coefficient of variation (CV) = (standard deviation/mean) x 100

$$CV = \frac{[\Sigma(X_i - \bar{X})^2/n - 1]^{1/2}}{\bar{X}} \times 100$$

where: X_i = individual measurement

n = number of replicates

$\bar{X}$ = mean of measurements

Table 7

PHOSPHATE METHODS COMPARISON

Method	Range	Interferences/ Sources of Bias	Applications	Preparation Time[a] (hr)	Sample Analysis Time[b] (hr/single sample)	Single Operator Coefficient of Variation[c] (%)	Special Equipment Requirements
Ion Chromatography 50% Modified Anion Eluent	8 to 23 mg/l in the aliquot injected	High sulfate-to-phosphate molar ratios inhibit phosphate peak resolution.	Liquors from low TDS lime or limestone systems where chloride and sulfate concentrations are low enough to allow peak resolution	3.25	0.25	20	Ion chromatograph with anion separator and suppressor columns Strip chart recorder
Vanadomolybdate Spectrophotometric	1.0 to 18 mg/l (Refer to Method 53.)	Arsenic and silica at 133 mg/L may cause positive interference. Ferrous iron (33 and 133 mg/L) at 400 nm (Use 470 nm.) Chloride at 75 mg/L (Use HCl in vanadate-molybdate reagent.) Only ortho form of phosphate ion is measured (Digest sample.)	Liquors from lime, limestone, dual alkali, and magnesia processes	4.5	0.35	2 (est.)	Spectrophotometer with 1 cm cuvettes

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[a] Includes labor hours for equipment set up, instrument calibration, reagent preparation and standardization.

[b] Elapsed time from receipt of one sample to calculated final result including sample preparation, analysis, and calculations. Clean up time is not included. Actual labor hours are less than elapsed time in some cases. Labor hours per sample will be reduced if more than one sample is analyzed.

[c] Coefficient of variation (CV) = (standard deviation/mean) x 100

- When preparing the regenerant solution and HCl (Section 10), be sure to wear safety glasses and work under a hood. Be sure to add acids to an excess of water and allow the solutions to cool before adding additional acid. These steps will prevent acid spattering which may cause severe burns.

9.0 EQUIPMENT AND REAGENTS

Apparatus

- Dionex Model 10, 14, or 16 Ion Chromatograph
- AS3 anion separator column
- AG3 anion guard column
- ASC-2 anion packed-bed suppressor column
- Strip chart recorder with input voltage settings covering the range of 0.1 to 1.0 volt. This voltage range is necessary to optimize the high standard peak height to 85-90% of the chart paper capacity
- Analytical balance accurate to 0.0001 g
- Volumetric flasks (at least one each 50 mL, 100 mL, 200 mL, 250 mL, 500 mL, and 1000 mL)
- Pipettes (1 each 2, 3, 4, 5, 6, 7, 8, and 10 mL for preparing standards and diluting samples)
- Syringes (luer tip, 3.0 mL capacity)
- Engineer's scale for measuring peak heights
- Weigh papers
- Spatula
- Source of air or compressed gas (80 psig minimum)
- Calculator with linear least squares function
- Plastic bottles
- Pipette bulb

- Magnetic stirrer, Corning PC-353 or equivalent
- Magnetic stir bar
- Oven, 120°C, for drying reagents
- Electric timer
- Glass graduated cylinders, 100 mL and 25 mL

Reagents (reagent-grade quality)

- Source of deionized water (conductivity <5 µmhos/cm)
- Sodium fluoride, NaF
- Sodium chloride, $NaCl$
- Sodium sulfite, Na_2SO_3
- Sodium sulfate, Na_2SO_4
- Potassium sulfate, K_2SO_4
- Disodium hydrogen phosphate, $Na_2HPO_4 \cdot 7H_2O$
- Sodium hydroxide, $NaOH$
- Sodium carbonate, Na_2CO_3
- Sulfuric acid, H_2SO_4, concentrated
- Hydrochloric acid, HCl, concentrated

Solutions

- Stock fluoride solution, 0.0026 M
- Stock chloride solution, 0.004 M
- Stock sulfite solution, 0.004 M
- Stock sulfate solution, 0.004 M
- Stock phosphate solution, 0.008 M
- Anion standard solutions

- Concentrated modified anion eluent (0.52 M NaOH and 0.70 M Na_2CO_3)
- Working modified anion eluent (0.0013 M NaOH + 0.00175 M Na_2CO_3)
- Regenerant solution (1.0 N H_2SO_4)
- Quality control sample, 0.001 M Cl^- + 0.001 M $SO_4^{=}$
- Hydrochloric acid (1.0 N)

10.0 SOLUTION/REAGENT PREPARATION

- Stock fluoride solution--Weigh 0.1092 g NaF. Transfer quantitatively to a 1-liter volumetric flask. Add deionized water, mix until all solids have dissolved, and dilute to volume. Store in an airtight polyethylene bottle.
- Stock chloride solution--Weigh 0.2338 g NaCl. Transfer quantitatively to a 1-liter volumetric flask. Add deionized water, mix, dilute to mark. Store in a 1-liter polyethylene or glass bottle.
- Stock sulfite solution--Weigh 0.5044 g Na_2SO_3. Transfer quantitatively to a 1-liter volumetric flask. Add deionized water and 0.02 M (0.6 mL) formaldehyde. Mix until all solids have dissolved. Dilute to volume with deionized water. Prepare fresh daily.
- Stock sulfate solution--Weigh 0.6972 g K_2SO_4. Transfer quantitatively to a 1-liter volumetric flask. Add deionized water, mix, then dilute to volume. Store in a 1-liter polyethylene or glass bottle.
- Stock phosphate solution--Weigh 2.8640 g $Na_2HPO_4 \cdot 7H_2O$. Transfer quantitatively to a 1-liter volumetric flask. Add deionized water, mix, and dilute to volume. Store in a 1-liter polyethylene or glass bottle. [Note: A recent Dionex users' memorandum suggested replacing disodium hydrogen phosphate with anhydrous potassium dihydrogen phosphate (KH_2PO_4) for preparation of phosphate standards. This change is recommended because the hydration value of the disodium hydrogen phosphate is uncertain, (2)].
- Anion standard solutions--Dilute stock solutions as indicated in Table 8.

Table 8

STANDARD SOLUTION PREPARATION GUIDE

	Fluoride		Chloride		Sulfite		Sulfate		Phosphate	
Dilution Volume (ml)	Pipet Volume (ml)	Resulting Concentration of Std (mg/L)	Pipet Volume (ml)	Resulting Concentration of Std. (mg/L)	Pipet Volume (ml)	Resulting Concentration of Std. (mg/L)	Pipet Volume (ml)	Resulting Concentration of Std. (mg/L)	Pipet Volume (ml)	Resulting Concentration of Std. (mg/L)
500	5	0.494	5	1.42	5	3.20	5	3.84	5	7.60
200	5	1.24	5	3.55	4	6.41	4	7.69	3	11.4
100	5	2.47	4	5.67	4	12.8	4	15.4	2	15.2
100	8	3.95	6	8.51	7	22.4	7	26.9	3	22.8

- Concentrated modified anion eluent--Weigh 20.8 g NaOH and 74.2 g Na_2CO_3. Transfer quantitatively to a 1-liter volumetric flask. Add deionized water, mix until all solids have dissolved, then dilute to volume. Store in a 1-liter polyethylene or glass bottle up to two months.

- Working anion eluent--Pipet 10 mL of concentrated modified anion eluent into a plastic Dionex reservoir for 50% strength modified anion eluent and fill to the 4-liter mark. Cap and shake vigorously before removing excess air space from the reservoir. Prepare fresh weekly.

- Regeneration solution--Carefully measure 111 mL concentrated H_2SO_4 by pouring into a glass graduated cylinder. Be sure to wear safety glasses. Work under a hood if possible. Slowly add the acid in 10 mL increments to approximately 3800 mL deionized water in a glass container in a cold water bath. Stir the solution with a glass rod between acid additions. Allow solution to cool before adding more acid. When all acid has been added and the solution has cooled to room temperature, transfer to an eluent reservoir bag. CAUTION: Be certain to add the acid to the water to prevent spattering of acid which will cause severe burns.

- Quality control sample--Weigh 0.0585 g oven-dried (2 hrs at 110°C) NaCl and 0.1421 g oven-dried Na_2SO_4. Transfer to a 1-liter volumetric flask. Add deionized water, mix to dissolve all solids, and dilute to volume. Store in a 1-liter polyethylene or glass bottle.

- 1.0 N hydrochloric acid—Carefully pour 21 mL concentrated HCl into a glass graduated cylinder. Be sure to wear safety glasses and work under a hood. Slowly add the acid to approximately 200 mL deionized water in a 250 mL volumetric flask. Allow solution to cool. Bring volume to the mark with deionized water. CAUTION: Be certain to add the acid to an excess of water (at least 200 mL) to prevent spattering of acid which can cause serious burns.

11.0 QUALITY ASSURANCE/QUALITY CONTROL

Results can be verified by duplicate analyses, analysis of QC standards, analysis of blanks, and analysis of spiked samples. These QC procedures should be implemented with the frequencies in Table 9.

Table 9

QUALITY CONTROL SAMPLE GUIDE

Number of Samples	Number of Full Sets of Standards	Number of Mid Range Standards	Number of Duplicate Analyses	Number of Blanks	Number of QC Standards	Number of Sample Spikes
1-10	2	1	2	1	1	1
11-20	3	2	3	1	1	2
21-40	3	2	4	2	2	3

The directions for preparation of standards and a QC sample are provided in Section 10. Limits should be set (based on precision of the method) for acceptable variations in duplicate measurements and in daily measurements on the QC sample. If measured concentrations fall outside acceptable limits, the possibility of an analytical problem is indicated and the source of the deviation should be identified. If an analytical problem is found, appropriate corrective action should be taken. Sample "spikes" are prepared by adding a known amount of the anion of interest to an aliquot of the sample. The results of analysis of the sample and the spiked sample are compared to determine the "recovery" of the known standard addition. If the amount recovered differs significantly from the amount added, it is concluded that the sample itself (the "matrix") has influenced the results of the analysis. The presence of interfering species should be suspected. If matrix interferences occur, either another analytical method can be used, or the method of standard addition can be applied to determine a correction.

The ion chromatograph should be calibrated for each sample set analyzed. This is accomplished by introducing standard solutions into the instrument according to the procedure outlined in Section 13. There should be a linear relationship between concentration and peak height. (The graph should be a straight line). A least squares curve fit technique should be used to determine the correlation coefficient, which indicates the amount of scatter of the calibration points around the line. Perfect correlation is indicated by a correlation coefficient of 1.0. A calibration line with a correlation coefficient of less than 0.995 is not valid, and a recalibration must be done. (NOTE: If it is obvious that one calibration point is an outlier, reanalyze that point and again check the validity of the calibration.)

12.0 SAMPLE ACQUISITION AND HANDLING

Samples collected from an FGD system can be unstable (supersaturated) and can contain volatile compounds (carbonate and sulfite). To stabilize the sample, the following procedures should be implemented:

1. Separate liquid and solid phases at sample ports using a peristaltic pump and in-line nucleopore filter.

2. Filter the liquor directly into a preweighed solution of deionized water containing formaldehyde at 5 times the molarity of the expected aqueous sulfite concentration.

Separation of liquids and solids will prevent dissolution or precipitation which would change the composition of the liquid or solid. Formaldehyde addition will inhibit sulfite oxidation to sulfate. Sulfite can then be measured directly in the same sample as the other ions, instead of oxidizing it to sulfate and measuring "total sulfur". If this sample acquisition procedure is followed, the samples will be ready for immediate analysis. They also can be stored for analysis at a later date without additional preservation.

When diluting samples, it is necessary to calculate a sampling dilution factor. This value should be used to correct the analytical results for the dilution effect. It can be calculated using the following equation:

$$DF = \frac{\frac{W_3 - W_2}{SG} + W_2 - W_1}{\frac{W_3 - W_2}{SG}}$$

where: DF = sampling dilution factor (dimensionless),

W_1 = weight of empty sample container (g),

W_2 = weight of sample container and dilution solution (g),

W_3 = weight of sample containter, dilution solution, and sample (g), and

SG = specific gravity of the sample.

If the specific gravity of the liquor sample is not known, a value of 1.0 may be used without serious error in many cases. The magnitude of the error will be greater the higher the dissolved solids concentration (refer to volume 1 of handbook). If a specific gravity of 1.00 is used, the dilution factor equation reduces to:

$$DF = \frac{W_3 - W_1}{W_3 - W_2}$$

Every attempt should be made to use the actual specific gravity for high TDS liquors since deviation of the sample density from 1 will cause error in the calculated dilution factor and all analytical results. Specific gravity can be measured easily using Method 10.

Solid samples must be prepared by the procedure outlined below.

Preparation of Solid Samples by Acid Dissolution

1. Weigh approximately 0.5 g of dry solids to the nearest 0.0001 g and record the weight.

2. Quantitatively transfer the weighed solids to a 250 mL volumetric flask containing approximately 100 mL water and a magnetic stir bar.

3. Add 3 mL 30% hydrogen peroxide to the flask, cap, and place on a magnetic stir plate. Stir for 5 minutes. The flask should be inverted intermittently during this time.

4. Add 20 mL of 1.0 N HCl, cap, and stir for 30-35 minutes.

5. After stirring, remove the stir bar from the flask and bring to volume with deionized water.

6. Assemble a filter apparatus containing a weighed filter membrane with a pore size of 0.45 µm.

7. Rinse the filter holder and filtration flask twice by drawing small aliquots of the sample through the filter and swirling in the filtration flask. Discard this filtrate. The remaining dissolution should then be filtered without rinsing either the volumetric flask or filter holder.

8. Transfer the filtrate to a clean, dry sample bottle and label accordingly.

9. Analyze for total sulfur as sulfate by ion chromatography using the method for liquid samples (Section 13).

10. The acid-insoluble ("inerts") portion of the solid sample can be measured by drying and weighing the filter plus solids obtained in Step 7.

13.0 STEPWISE PROCEDURE

Instrument Set-Up

1. Turn on the ion chromatograph power switch.

2. Turn on the air to the ion chromatograph, at least 80 psi, and check to be certain the air line is free of water.

3. Check to be certain all eluent reservoirs are open, and that the DI water reservoir is full.

4. Start the eluent pump on deionized water and flush the plumbing system for 10 minutes at 33% flow.

5. Change to the anion eluent and flush the plumbing system 10 minutes.

6. Switch the separator and suppressor columns in line.

7. Check the pressure; if it is greater than 650 psi, reduce the flow rate.

8. Visually inspect the columns and fittings for leaks.

9. If system pressure is below 150 psi with columns in line, switch columns out of line and disconnect the pump's bubble trap inlet to allow air bubbles to escape. Reconnect the bubble trap and switch columns back in line.

10. Allow the system to stabilize for about 15 minutes.

11. Set full-scale on the conductivity meter to 30 μmhos.

12. Turn the mode switch to linear.

13. Set the conductivity to zero using the offset control.

14. Turn on the recorder at 0.5 V input voltage and 30 cm/hr chart speed.

15. Adjust the recorder zero to 5% of full scale.

16. Watch the recorder trace for a stable baseline.

17. Reset the offset control if necessary.

18. Analyses can begin when the baseline is stable (5-10 minutes).

19. When peaks do not return to baseline before elution of the next species, baselines should be drawn in as in Figure 1.

Instrument Calibration

1. Set the inject/load switch to the load position.

2. Flush a 3 mL syringe with the highest concentration standard.

3. Flush the sample loop with 3 mL of the highest standard.

4. Introduce a second 3 mL portion and flip the switch to the inject mode. Press the pip button to mark the point of injection on the chart paper.

5. After a minute or two, flip the injection switch back to the load position.

6. Watch the recorder trace, noting the elution time of the peaks and the height of the peaks. If the peak is less than 1/2 of the chart paper in height, switch the recorder to 0.2 V input; if the peak is off scale, switch the input voltage to one volt. Some recorders have a fine adjustment control which allows the peak height to be adjusted more precisely. 85-90% of the chart height is a good height for the high standard.

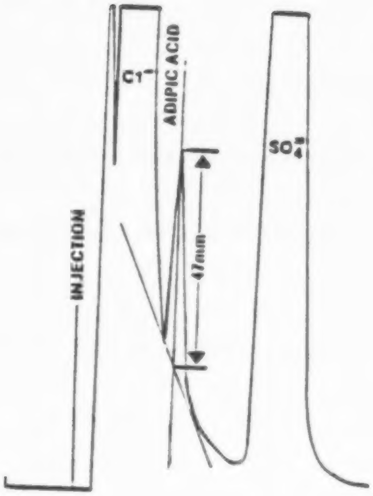

Figure 1. Peak Height Measurement on a Partially Resolved Ion Chromatography Peak

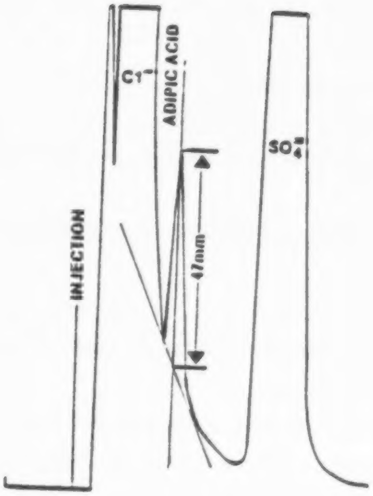

Figure 1. Peak Height Measurement on a Partially Resolved Ion Chromatography Peak

7. Repeat Steps 2 through 5 for the other standards and a blank.

8. From the instrument response (peak height) during analysis of the blanks and calibration standards, determine the correlation coefficient of the calibration curve using a least square curve fit technique (refer to volume 1 of handbook). An acceptable calibration curve should have a correlation coefficient of no less than 0.9950. Or, plot the calibration curve on a sheet of linear graph paper. Plot the standard concentration (mg/L) on the x-axis (horizontally) and peak height on the y-axis (vertically). After the four points (one per standard) have been marked on the graph, draw a straight line passing as close as possible to every point. This line represents the standard curve. To determine the concentration of an analyzed sample, find its peak height on the vertical axis, move to the right in a straight line until you reach the standard curve, then read the concentration from the horizontal axis directly below the point on the standard curve. If the linear least squares curve fit technique has been used, the concentration in the unknown sample is calculated from the peak height and the slope and intercept of the calibration line (refer to volume 1 of handbook).

Sample Analysis

1. Once an acceptable calibration has been obtained, repeat Steps 2 through 5 described above for instrument calibration for the samples, QC standards and spiked samples.

2. If necessary, dilute the sample so that it is in the same concentration range as the standards. Record both the original sample volume, V_P, and the diluted volume, V_T, on the data recording form (Figure 3, Section 14). If the concentrations of the anions in a sample vary widely, it may be necessary to perform a separate dilution and injection for each anion. In typical scrubber liquors, chloride and sulfate can be analyzed in one dilution, and another is required for fluoride, sulfite and phosphate.

3. When analyzing samples, wait until all peaks have eluted before injecting the next sample.

4. Record the sample number or standard identification and sampling dilution factor, DF, on the strip chart by the pip mark and on the data recording form.

5. Run quality control samples as required (see Table 9). Quality control samples should be interspersed with other samples.

Instrument Shutdown

1. Turn the mode switch to zero and switch the eluent to water. Run water through the anion system for 20 minutes.

2. Bypass both columns by turning the switches to the down position.

3. Turn the eluent pump off and empty the waste container as necessary.

4. If the unit has been run for 8 to 10 hours, regenerate the suppressor column.

Regeneration of the Suppressor Column

1. Make up 4 liters of 1 N H_2SO_4 regenerant solution according to the directions in Section 10. Rinse a 4-liter reservoir with several small aliquots of the regenerant. Fill the reservoir with the regenerant and attach to the system regenerant connection. Open the reservoir.

2. Set regeneration time at 15 minutes and the rinse time at 35 minutes.

3. Press the green "start" button. The instrument will run through the regeneration cycle and turn off automatically. The air must be left on until the regeneration cycle is completed.

14.0 DATA RECORDING FORM

Figures 2, 3, and 4 show data recording forms for instrument calibration and liquid and solid sample analysis.

15.0 DATA REDUCTION AND CALCULATIONS

Accurately measure peak heights on chart paper using an engineer's scale. (The scale with 50 subdivisions per inch should be used.) Record the number of height units for each standard or sample on the data recording form.

Use the peak heights for standards and blanks (Figure 2) to plot a calibration line, or use a linear least square curve fit technique to calculate slope, intercept and correlation coefficient.

Laboratory Name: ______________________ Analyst Initials: ______________

Analysis Date: ______________________ Instrument: ______________

Standard I.D.	Analyte	Theoretical Injected Concentration (mg/L)	Peak Height	Calibration Data			Instrument Conditions			
				Slope	Intercept	Correlation Coefficient	Offset	Voltage Input	Flow Rate	µmhos cm
1.										
2.										
3.										
4.										

Figure 2. Ion Chromatography Calibration Data Recording Form.

Laboratory Name: ______________________ **Analyst Initials:** ______________________

Analysis Date: ______________________ **Instrument:** ______________________

Sample I.D.	Analyte	Peak Height	C_G Concentration In Injected Sample (mg/L)	DF Sampling Dilution Factor	V_P Original Sample Volume (mL)	V_T Diluted Sample Volume (mL)	C_L Concentration In Original Liquid Sample (mg/L)
1.							
2.							
3.							
4.							

$$C_L = C_G \left(\frac{V_T}{V_P}\right) DF$$

Figure 3. Ion Chromatography Data Recording Form for Liquid Samples.

Laboratory Name: ____________________ Analyst Initials: ____________________

Analysis Date: ____________________ Instrument: ____________________

Sample I.D.	Analyte	Peak Height	C_G Concentration In Injected Sample (mg/L)	V_S Dissolution Volume (mg/L)	W Weight of Solid Sample (g)	C_S Concentration In Original Solid Sample (mg/g)
1.						
2.						
3.						
4.						

$$C_S = \frac{V_S C_G}{1000\ W}$$

Figure 4. Ion Chromatography Data Recording Form for Solid Samples.

For each sample peak height recorded on Figure 3 or 4, read the anion concentration in the injected sample, C_G, from the calibration plot, or calculate it from the slope and intercept of the calibration line.

Calculate the concentration in the original liquid sample using the following equation:

$$C_L = C_G \left(\frac{V_T}{V_P}\right) DF$$

where: C_L = concentration in original liquid (mg/L),
C_G = concentration in injected sample read or calculated from calibration data (mg/L),
V_T = volume sample was diluted to in the laboratory (mL),
V_P = volume of original sample taken for analysis (mL), and
DF = sampling dilution factor calculated as described under "Sample Acquisition and Handling" (dimensionless).

Calculate the concentration in the original solid sample using the following equation:

$$C_S = \frac{C_G V_S}{1000 W}$$

where: C_S = concentration in the original solid (mg/g),
C_G = concentration in injected sample from calibration data (mg/L),
V_S = dissolution volume (volume to which original solid sample was dissolved, mL), and
W = weight of solid sample (g).

The 1000 factor in the denominator is used to convert mL to L. To express the results for solid samples in weight percent, divide C_S by 10.

16.0 PRECISION AND BIAS

The precision and bias of this ion chromatography procedure have been investigated (refer to volume 1 of handbook). The procedure given in Section 13 was used to analyze fluoride, chloride, sulfite, sulfate, and phosphate in synthetic scrubber liquors and total sulfur in solids obtained from two operating lime FGD systems. The synthetic liquors were prepared from reagent grade chemicals with compositions typical of scrubbing liquors from lime, limestone, magnesia and dual alkali scrubbing processes. The linear least squares curve fit technique was used to prepare calibration curves.

Table 10 gives the results of determination of single operator precision and bias for fluoride analysis in five synthetic scrubber liquors, all containing about the same level (4-10 mg/L) of fluoride. The pooled coefficient of variation for all materials tested was 14.9 percent. The percent bias based on known amounts of fluoride added to the solutions ranged from -12 to +72 percent. Statistically significant bias was found for all types of scrubber liquors.

Fluoride peak resolution is affected by high sulfate and chloride concentrations. Table 11 shows how both precision and bias are affected by high chloride-to-fluoride and high sulfate-to-fluoride mole ratios. The highest coefficients of variation and the largest bias were found for solutions with high sulfate-to-fluoride and/or high chloride-to-fluoride ratios.

The results in Table 11 indicate that care should be used in applying this method to liquors from high TDS limestone, dual alkali, or magnesia scrubbing systems which typically contain high levels of sulfate and chloride.

Table 12 gives the results of determination of single operator precision and bias for chloride analysis in five synthetic scrubber liquors and a standard made from sodium chloride in deionized water. The pooled coefficient of variation for all materials tested is 12.3 percent. The percent bias based on known amounts of chloride added to the solutions ranged from -4.8 to 8.4

Table 10

RESULTS OF INVESTIGATION OF PRECISION AND BIAS OF FLUORIDE ANALYSIS BY ION CHROMATOGRAPHY WITH MODIFIED ANION EFFLUENT

Sample ID and Description[1]	True Value[2] (mg/L)	n = number of Replicates[3]	Mean of Measurements (mg/L)	Precision[4] Standard Deviation (mg/L)	Precision[4] Coefficient of Variation (%)	Bias[5] (%)
LQ5-High TDS limestone scrubber liquor	3.95	8	5.3	0.91	17.	34.
LQ4-Low TDS limestone scrubber liquor	4.94	8	4.8	0.09	1.9	-3.4
LQ1-High magnesium lime scrubber liquor	5.93	8	5.2	0.19	3.6	-12
LQ2-Magnesia (high Mg) scrubber liquor	7.98	8	13.7	3.8	27.	72
LQ3-Dual alkali scrubber liquor	9.88	8	12.4	1.1	8.6	26

---

[1] Sample ID

	Composition (mg/L)							
	Ca	Mg	Na	SO_4	CO_3	Cl	F	SO_3
LQ1	400	1,215	385	6,144	30	284	5.93	80
LQ2	160	36,500	1,200	146,000	120	285	7.98	
LQ3	40	122	45,800	48,000	36,000	14,300	9.88	
LQ4	561	243	647	2,570	300	600	4.94	160
LQ5	20,000	608	238	768	6	35,800	3.95	

[2] Known amount of reagent grade chemicals added to deionized water

[3] Number of times a separate sample of the same material was analyzed by the same analyst with the same instrument and a separate calibration

[4] Standard deviation = $[\sum (X_i - \bar{X})^2/n-1]^{1/2}$

Coefficient of variation = (standard deviation/mean) x 100

[5] Bias = [(mean - true value)/true value] x 100

*Not statistically significant at the 0.95 level of confidence

Table 11

EFFECT OF CHLORIDE AND SULFATE ON PRECISION AND BIAS OF ANALYSIS OF 4-10 MG/L FLUORIDE IN SYNTHETIC SCRUBBER LIQUORS

Scrubber Liquor	Mole Sulfate / Mole Fluoride	Mole Chloride / Mole Fluoride	Coefficient of Variation (%)	Bias (%)
LQ1-High magnesium lime scrubber liquor	200	26	4	-12
LQ2-Magnesia (high Mg) scrubber liquor	2,000	9	27	72
LQ3-Dual alkali scrubber liquor	1,000	800	9	26
LQ4-Low TDS limestone scrubber liquor	100	60	2	-3.4
LQ5-High TDS limestone scrubber liquor	40	5,000	17	34

Table 11

EFFECT OF CHLORIDE AND SULFATE ON PRECISION AND BIAS OF ANALYSIS OF 4-10 MG/L FLUORIDE IN SYNTHETIC SCRUBBER LIQUORS

Scrubber Liquor	Mole Sulfate / Mole Fluoride	Mole Chloride / Mole Fluoride	Coefficient of Variation (%)	Bias (%)
LQ1-High magnesium lime scrubber liquor	200	26	4	-12
LQ2-Magnesia (high Mg) scrubber liquor	2,000	9	27	72
LQ3-Dual alkali scrubber liquor	1,000	800	9	26
LQ4-Low TDS limestone scrubber liquor	100	60	2	-3.4
LQ5-High TDS limestone scrubber liquor	40	5,000	17	34

Table 12

RESULTS OF INVESTIGATION OF PRECISION AND BIAS OF CHLORIDE ANALYSIS BY ION CHROMATOGRAPHY WITH MODIFIED ANION ELUENT

Sample ID and Description[1]	True Value[2] (mg/L)	n = number of Replicates[3]	Mean of Measurements (mg/L)	Precision[4] Standard Deviation (mg/L)	Precision[4] Coefficient of Variation (%)	Bias[5] (%)
QCAB-NaCl in deionized water	35.5	8	34.0	1.4	4.2	1.9*
LQ1-High magnesium lime scrubber liquor	284.	7	276	4.6	1.7	-2.7
LQ2-Magnesia (high Mg) scrubber liquor	285	8	309	84	27.	8.4*
LQ4-Low TDS limestone scrubber liquor	603	8	575	61	11	-4.7*
LQ3-Dual alkali scrubber liquor	14,300	8	13,600	543	4.0	-4.8
LQ5-High TDS limestone scrubber liquor	35,800	8	37,200	854	2.3	3.9

---

[1] Sample ID

	Composition (mg/L)					
	Ca	Mg	Na	SO_4	CO_3	Cl
LQ1	400	1,215	379	6,144	30	284
LQ2	160	36,500	1,200	146,000	120	285
LQ3	40	122	45,800	48,000	36,000	14,300
LQ4	561	243	643	2,570	300	600
LQ5	20,000	608	237	768	6	35,800

[2] Known amount of reagent grade chemicals added to deionized water

[3] Number of times a separate sample of the same material was analyzed by the same analyst with the same instrument and a separate calibration

[4] Standard deviation = $[\sum (X_i - \bar{X})^2/n-1]^{1/2}$

Coefficient of variation = (standard deviation/mean) x 100

[5] Bias = [(mean - true value)/true value] x 100

* Not statistically significant at the 0.95 level of confidence

percent. No statistically significant bias was found for the sodium chloride sample, the low TDS limestone or the magnesia scrubbing liquor. The highest bias and coefficient of variation were found for the high magnesium scrubbing liquor, which has the highest sulfate to chloride ratio of the solutions tested. However there does not appear to be a significant correlation between sulfate to chloride ratio and precision and bias in the other samples.

Table 13 shows the single operator precision and bias for sulfite analysis in the five liquors and a sample prepared from sodium sulfite in deionized water. The sulfite concentration in the samples ranged from 8 to 40,000 mg/L. The pooled coefficient of variation for all materials tested is 11.4 percent, but this is a misleading precision limit since the precision is concentration dependent. The pooled coefficient of variation for solutions containing 80 to 40,000 mg/L sulfite is 2.5 percent.

The percent bias based on known amounts of sulfite added ranged from -1.7 to 72 percent. The bias was not statistically significant for the two samples with the highest sulfite concentrations. Bias was positive (indicating high recovery) for all samples with significant bias.

Table 14 shows that precision and bias clearly depend on sulfite concentration, and that the method is much more accurate when applied to liquors with high sulfite concentrations. High sulfate concentration hinders sulfite peak separation and contributes to the poor accuracy in low-sulfite liquors. This method is not accurate for sulfite analysis in high TDS or forced oxidized systems where sulfate levels are much higher than sulfite levels. It is applicable in high magnesium lime, magnesia, dual alkali and low TDS limestone systems.

Table 15 gives results of determination of the single operator precision and bias for sulfate analysis in five scrubber liquors and a sample prepared from potassium sulfate in deionized water. The sulfate concentration in the scrubber liquors ranged from 770 to 146,000 mg/L. The pooled coefficient of variation for all materials tested is 3.7 percent. The percent

Table 13

RESULTS OF INVESTIGATION OF PRECISION AND BIAS OF SULFATE ANALYSIS BY ION CHROMATOGRAPHY WITH MODIFIED ANION ELUENT

Sample ID and Description[1]	True Value[2] (mg/L)	n = number of Replicates[3]	Mean of Measurements (mg/L)	Precision[4] Standard Deviation (mg/L)	Precision[4] Coefficient of Variation (%)	Bias[5] (%)
LQ5-High TDS limestone scrubber liquor	8.01	5	13.8	4.4	32.	72
QCAB-Sodium sulfite in deionized water	9.88	8	12.4	1.1	8.7	26
LQ1-High magnesium lime scrubber liquor	80.1	8	88.7	2.7	3.0	11
LQ4-Low TDS limestone scrubber liquor	160	7	171	5.1	3.0	6.4
LQ2-Magnesia (high Mg) scrubber liquor	2,400	7	2,430	53	2.2	0.9*
LQ3-Dual alkali scrubber liquor	39,700	8	39,000	860	2.2	-1.7*

---

[1] Sample ID — Composition (mg/L)

Sample ID	Ca	Mg	Na	SO_4	CO_3	Cl	F	SO_3	PO_4
LQ1	400	1,215	385	6,144	30	284	5.93	80	0
LQ2	160	36,500	1,200	146,000	120	285	0	2,400	0
LQ3	40	122	45,800	48,000	36,000	14,300	0	39,700	0
LQ4	561	243	647	2,570	300	600	4.94	160	53.2
LQ5	20,000	608	239	768	6	35,800	0	8.01	0

[2] Known amount of reagent grade chemicals added to deionized water

[3] Number of times a separate sample of the same material was analyzed by the same analyst with the same instrument and a separate calibration

[4] Standard deviation = $[\sum (X_i - \bar{X})^2/n-1]^{1/2}$

Coefficient of variation = (standard deviation/mean) x 100

[5] Bias = [(mean - true value)/true value] x 100

*Not statistically significant at the 0.95 level of confidence

Table 14

EFFECT OF SULFITE CONCENTRATION AND SULFATE-SULFITE MOLE RATIO ON PRECISION AND BIAS OF SULFITE ANALYSIS BY ION CHROMATOGRAPHY

Sulfite Concentration (mg/L)	$\frac{\text{Moles SO}_4}{\text{Moles SO}_3}$	Coefficient of Variation (%)	Bias (%)
8	80	32	72
10	0	9	26
80	64	3	11
160	14	3	6
2,400	50	2	0.9*
40,000	1	2	-1.7*

---

*Not statistically significant

Table 15

RESULTS OF INVESTIGATION OF PRECISION AND BIAS OF SULFATE ANALYSIS BY ION CHROMATOGRAPHY WITH MODIFIED ANION ELUENT

Sample ID and Description[1]	True Value[2] (mg/L)	n = number of Replicates[3]	Mean of Measurements (mg/L)	Precision[4] Standard Deviation (mg/L)	Precision[4] Coefficient of Variation (%)	Bias[5] (%)
QCAB-sodium sulfate in deionized water	96.1	8	95.4	2.9	3.0	-0.8*
LQ5-High TDS limestone scrubber liquor	768	8	745	50	6.6	-3.0*
LQ4-Low TDS limestone scrubber liquor	2,570	8	2,800	70	2.5	8.8
LQ1-High magnesium lime scrubber liquor	6,150	8	6,110	47	0.8	-0.6*
LQ3-Dual alkali scrubber liquor	48,000	8	47,500	1,800	3.8	-1.0*
LQ2-Magnesia (high Mg) scrubber liquor	146,400	8	146,400	4,500	3.1	0*

---

[1] Sample ID

	Composition (mg/L)					
	Ca	Mg	Na	SO_4	CO_3	Cl
LQ1	400	1,215	379	6,144	30	284
LQ2	160	36,500	1,200	146,000	120	285
LQ3	40	122	45,800	48,000	36,000	14,300
LQ4	561	243	643	2,570	300	600
LQ5	20,000	608	237	768	6	35,800

[2] Known amount of reagent grade chemicals added to deionized water

[3] Number of times a separate sample of the same material was analyzed by the same analyst with the same instrument and a separate calibration

[4] Standard deviation = $[\sum (X_i - \bar{X})^2/n-1]^{1/2}$
Coefficient of variation = (standard deviation/mean) x 100

[5] Bias = [(mean - true value)/true value] x 100

* Not statistically significant at the 0.95 level of confidence

bias based on known amounts of sulfate added to the solutions ranged from -3 to 8.8; however, statistically significant bias was found only for the low TDS limestone scrubber liquor. The cause of the unusually high bias for this sample is not apparent.

Table 16 shows single operator precision and bias for phosphate analysis in two scrubber liquors at a level of 50 to 60 mg/L. Phosphate potentially could be present in scrubber liquors if cooling water blowdown were used for make up in closed loop systems. The level to which phosphate would build up is limited by the solubility of its calcium and magnesium salts, but there are no data to indicate typical levels in scrubber liquors. (The phosphate concentration in natural waters is on the order of 30 mg/L.)

Phosphate peak resolution was not possible in synthetic scrubber liquors with high chloride and/or sulfate concentrations. Resolution was achieved in the high-magnesium lime liquor and the low TDS limestone liquor. The pooled coefficient of variation for both materials was 20 percent. The bias was not statistically significant for either sample.

Table 17 shows precision data for analysis of total sulfur (sulfite plus sulfate) in solid samples containing 20 to 50 weight percent total sulfate (200-500 mg SO_4/g). The samples were obtained from two lime FGD systems, one employing a scrubber and the other a spray dryer. They were dissolved and oxidized using the procedure in Section 12 and analyzed using the procedure in Section 13. The single operator coefficient of variation for both materials is 4.1 percent. Therefore, the results of two properly conducted analyses by the same analyst on the same material should not differ by more than 11.6 percent of their average.

Table 18 summarizes the precision limits for all anions in samples containing calcium, magnesium, sodium, chloride, fluoride, phosphate, sulfite and sulfate. The results of two properly conducted analyses for an anion by

Table 16

RESULTS OF INVESTIGATION OF PRECISION AND BIAS OF PHOSPHATE ANALYSIS BY ION CHROMATOGRAPHY WITH MODIFIED ANION ELUENT

Sample ID and Description[1]	True Value[2] (mg/L)	n = number of Replicates[3]	Mean of Measurements (mg/L)	Precision[4] Standard Deviation (mg/L)	Precision[4] Coefficient of Variation (%)	Bias[5] (%)
LQ4-Low TDS limestone scrubber liquor	60.8	8	61.4	4.7	7.6	1.0*
LQ1-High magnesium lime scrubber liquor	53.2	8	57.0	15	27	7.3*

---

[1] Sample ID

	Composition (mg/L)								
	Ca	Mg	Na	SO_4	CO_3	Cl	F	SO_3	PO_4
LQ1	400	1,215	385	6,144	30	284			60.8
LQ2	160	36,500	1,200	146,000	120	285	7.98	2400	0
LQ3	40	122	45,800	48,000	36,000	14,300	9.88	39700	0
LQ4	561	243	647	2,570	300	600	4.94	160	53.2
LQ5	20,000	608	238	768	6	35,800	3.95	8.07	0

[2] Known amount of reagent grade chemicals added to deionized water

[3] Number of times a separate sample of the same material was analyzed by the same analyst with the same instrument and a separate calibration

[4] Standard deviation = $[\sum (X_i - \bar{X})^2/n-1]^{1/2}$

Coefficient of variation = (standard deviation/mean) x 100

[5] Bias = [(mean − true value)/true value] x 100

*Not statistically significant at the 0.95 level of confidence

Table 17

RESULTS OF INVESTIGATION OF PRECISION OF TOTAL SULFUR ANALYSIS IN SCRUBBER SOLIDS BY ION CHROMATOGRAPHY WITH MODIFIED ANION ELUENT

Sample ID and Description	n = number of Replicates[1]	Mean of Measurements (mg/g)	Precision[2] Standard Deviation (mg/g)	Precision[2] Coefficient of Variation (%)
SL1-Lime spray dryer solids with 80% fly ash	8	226	11	5.1
SL1-Lime scrubber solids without fly ash	8	508	14	2.8

---

[1] Number of times a separate sample was analyzed by the same analyst with the same instrument and a separate calibration

[2] Standard deviation = $[\sum (X_i - \bar{X})^2/n-1]^{1/2}$

Coefficient of variation = (standard deviation/mean) x 100

Table 18

SUMMARY OF PRECISION LIMITS FOR ANALYSIS OF ANIONS BY ION CHROMATOGRAPHY WITH MODIFIED ANION ELUENT

Anion	Concentration Range (mg/L)	One Sigma Limit (Single Operator Coefficient of Variation) (1S%)	Difference Two Sigma Limit (D2S%)
Fluoride	4-10	14.9	42.2
Chloride	36-36,000	12.3	34.8
Sulfate	100-146,000	3.7	10.5
Sulfite	80-40,000	2.5	7.1
Phosphate	50-60	19.0	53.8

---

[1] These numbers represent the (1S%) and (D2S%) limits as described in ASTM Practice C670-81 for preparing precision statements for test methods for construction materials.

the same analyst on the same material using the same instrument should not differ by more than the percent of their average listed in the last column of Table 18.

17.0 REFERENCES

1. A. E. Behl. Radian Laboratory Notebook No. 000181. September 1982.
2. Ion Chromatograph Models 10 and 14 Operation and Maintenance Manual. Sunnyvale, CA: Dionex Corporation, 1979.
3. J. C. Terry and E. E. Ellsworth. IC Analysis of Formaldehyde Stabilized Sulfite Solutions. Radian Corporation In-House Study Project DCN 80-290-403-07-12-01, October 1980.
4. L. J. Holcombe and F. B. Meserole. Applications of Ion Chromatography for the Analysis of Utility Waste Liquors and Leachates. Radian Corporation paper presented at the 35th ACS Southwest Regional Meeting, December 1979.
5. L. J. Holcombe and J. C. Terry. Application of Ion Chromatography for the Analysis of Lime/Limestone Based SO_2 Scrubber Solutions. Radian Corporation Technical Note DCN 78-320-403-02, March 1978.

44. Analysis of Carbonate in Scrubber Liquor, Scrubber Solids and Limestone by Non-Dispersive Infrared (NDIR) Absorption Spectrophotometry

1.0 DESCRIPTION OF METHOD

A sample containing carbonate in aqueous solution is injected into an acid pool reactor where CO_2 is formed. The CO_2 is carried by a purge gas stream (N_2) to an absorption cell detector. The detector compares the absorbance of infrared radiation by the CO_2 from the sample with that of an identical cell containing pure N_2 at the frequency specific for CO_2.

The procedure outlined here is for an Oceanography International Total Carbon Analyzer. If another analyzer is used, consult the instrument manual for operating instructions.

2.0 SIGNIFICANCE AND USE

This method can be used to determine carbonate in limestone and solid and liquid FGD samples. It was developed as a "referee" method in an early study of analytical methods for lime/limestone scrubbing (3). The method is elaborate and requires time-consuming equipment calibration, solids preparation and standards preparation. However it is the best method for carbonate analysis if many samples are to be analyzed and carbonate (rather than total alkalinity) must be measured.

3.0 DETECTION LIMIT AND RANGE OF METHOD

Detection limit: 1 µg CO_3

Applicable range of procedure: 2 to 10 µg CO_3

4.0 ELAPSED TIME AND LABOR HOURS REQUIRED

Elapsed time from sample receipt to final answer: 5.5 hr (as indicated below assuming instrument is warm)

Instrument warm-up, span and zero adjustment: 5 hr

Syringe calibration: 1 hr/week

Operator/analyst time: 1.6 hr (not including reagent or standards preparation)

Activity	Elapsed Time (Hours)	Analyst Labor (Hours)
Standards/solution reagent preparation	2.0	2.0
Solid sample preparation	2.5	0.50
Instrument calibration	0.75	0.75
Sample analysis	0.10	0.10
Calculations	0.20	0.20

5.0 DEFINITIONS

infrared absorption spectrophotometry--involves the twisting, binding, rotating, and vibrating motions of atoms in a molecule. Incident radiation is absorbed at specific wavelengths depending on the functional groups in the molecule. The amount of radiation absorbed is proportional to the concentration.

acid pool reactor--a small ampule or chamber fitted with a septum and having an opening through which a stream of nitrogen is introduced. The ampule is filled with an acid mixture and injected with a sample containing carbonate. The liberated carbon dioxide is swept out by the nitrogen stream.

6.0 INTERFERENCES

SO_2, NO, NO_2 and H_2O all absorb radiation at the same wavelength as CO_2, so provisions are made to eliminate interference from these species. Hydrogen peroxide is added to the solution in the acid pool reactor to oxidize sulfite to sulfate. Phosphoric acid and potassium dihydrogen phosphate prevent stripping of NO and NO_2 (but not CO_2) if nitrite or nitrate are present. Water vapor is removed by adsorption on anhydrone.

7.0 ALTERNATE METHODS

A wet chemical method for carbonate (CO_2 evolution and absorption) is also included in this handbook. The choice of method depends on the time required, available equipment, and the required accuracy. These elements are summarized in Table 1 for the carbonate methods included in this handbook.

8.0 PRECAUTIONS/CRITICAL STEPS

The pH of the solution in the acid pool reactor must be less than about 5.6 for the complete reaction of carbonate and liberation/stripping of CO_2 to occur. The ideal operating pH is 4.3. Methyl red indicator is used to monitor the pH in the acid pool reactor. This indicator is red at pH 4.2,

Table 1

CARBONATE METHODS COMPARISON

Method	Range	Interferences/ Sources of Bias	Applications	Preparation Time[a] (hr)	Sample Analysis Time[b] (hr/single sample)	Single Operator Coefficient of Variation[c] (%)	Special Equipment Requirements
NDIR Absorption Spectrophotometry	2 to 10 μg CO_2	Sulfite (oxidize with peroxide) Nitrate and nitrite (add phosphoric acid and KH_2PO_4) Water vapor (adsorb onto anhydrone)	Limestones; solid and liquid FGD samples	5.25	0.30	Liquors 4.0 Limestones 5.3 Scrubber Solids 8.4	Non-dispersive infrared analyzer with integrator Standardization vial Syringes
CO_2 Evolution Barium Hydroxide Absorption	2,000 to 20,000 μg CO_2	Nitrate (acidify to pH 3) Nitrite (add sulfonic acid at pH 2.5) Sulfite (oxidize with peroxide)	Limestones; solid and liquid samples from lime, limestone, magnesia, and dual alkali FGD systems	2.0	1.2	Liquors (120-36,000 mg/l) 4.8 Limestones 2.7 Scrubber Solids 9.0	Burets Special glassware (refer to Equipment and Reagents Section)
Alkalinity by Acid/Base Titration	2 to 100 percent $CaCO_3$	Sulfite (oxidize with peroxide)	Carbonate in limestones; total alkalinity in scrubber solids	3.0	0.7	Synthetic Liquors 3.8 Limestones 2.0 Scrubber Solids 7.5	Buret

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[a] Includes labor hours for equipment set up, instrument calibration, reagent preparation and standardization

[b] Elapsed time from receipt of one sample to calculated final result including sample preparation, analysis, and calculations. Clean-up time is not included. Actual labor hours are less than elapsed time in some cases. Labor hours per sample will be reduced if more than one sample is analyzed.

[c] Coefficient of variation (CV) = (standard deviation/mean) x 100

light orange at pH 5.6 and yellow at pH 6.2. When the solution in the acid pool reactor is spent, it appears light orange and must be replaced.

The length of the digestion/dissolution step for solid samples is critical. A study of precision and bias of this method with four limestone samples and a sample of reagent grade calcium carbonate showed that a sample digestion period of 10 minutes was inadequate. Carbonate recovery was 15 to 21 percent low compared to recovery obtained with a 60 minute digestion period.

A digital integrator or a strip chart integrator should be used to determine peak area. Peak height is not an accurate measure of instrument response, since it varies with pH of the solution in the acid pool reactor. Peak area is independent of the acid pool pH.

9.0 EQUIPMENT AND REAGENTS

Apparatus

- Oceanography International Total Carbon System (or equivalent) non-dispersive infrared analyzer with digital or strip chart integrator
- Standardization vial (OIC No. M 125) used as acid pool reactor and for syringe calibration
- Analytical balance, accurate to 0.0001 g
- Hamilton Syringe No. CR-700-200 (or equivalent), 200 µL capacity
- 25 mL syringe
- Volumetric glassware (pipettes and flasks)
- Drying tubes and glass wool
- Ultrasonic bath (optional)

- 40 mL disposable septum sample bottles for dissolving solid samples (bottles - Wheaton No. 224833, caps - Wheaton No. 240517, disposable liners - Wheaton No. 240587)
- Oven for drying reagents 400°C
- Glass graduated cylinder, 10 mL

Reagents

- Sodium carbonate, Na_2CO_3
- Hydrogen peroxide, H_2O_2
- Potassium dihydrogen phosphate, KH_2PO_4
- Phosphoric acid, H_3PO_4
- Methyl red indicator
- Ammonium hydroxide, NH_4OH
- Anhydrous magnesium perchlorate, (anhydrone) $Mg(ClO_4)_2$
- Ammonium chloride, NH_4Cl
- Disodium EDTA, Na_2EDTA
- Hydrochloric acid, HCl (concentrated, 12.1 N)

10.0 SOLUTION/REAGENT PREPARATION

- Standard CO_2 solution, 100 mg/L--Dry approximately 0.5 g reagent grade anhydrous Na_2CO_3 at 400°C for at least 1 hour. Dissolve 0.0442 g in CO_2 free water and dilute to 250 mL in a volumetric flask. Store in an inverted flask fitted with an injection stopper and protected with ascarite (soda-asbestos) or soda-lime tubes.
- Potassium dihydrogen phosphate solution, 10%--Dissolve 100 g reagent grade KH_2PO_4 in deionized water and dilute to one liter in a volumetric flask. This solution is a component of the acid pool reactor solution.
- Methyl red indicator--Dissolve 0.1 g crystalline methyl red indicator in 100 mL deionized water. This indicator is added to the acid pool reactor solution.

- Anhydrous magnesium perchlorate--This material is used as a desiccant in the drying tubes.

- Buffer preservative solution--Dissolve 35.0 g NH_4Cl in 285 mL fresh NH_4OH and dilute to 500 mL with deionized water. Weigh 365 g Na_2EDTA in a separate container and add to the NH_4OH-NH_4Cl solution. This makes approximately 700 mL. This solution is used to preserve both liquid and dissolved solid samples.

- Phosphoric acid solution, 5%--Dilute 50 mL concentrated H_3PO_4 to one liter with deionized water. CAUTION: Always add concentrated acid to an excess of water, never the reverse. Use extreme caution when handling this or any other acid. The phosphoric acid is used in the acid pool reactor.

- Hydrochloric acid, 1 N--Measure 9.3 mL concentrated (12.1 N) HCl in a graduated cylinder. Slowly add to 100 mL of CO_2-free deionized water. This solution is used to dissolve solid samples.

- Acid reactor working solution--Add to 95 mL of 10% KH_2PO_4 solution, 5 mL 5% H_3PO_4, 2 mL 30% H_2O_2, and several drops of methyl red indicator.

11.0 QUALITY ASSURANCE/QUALITY CONTROL

Results of this procedure can be verified by implementing the following QA/QC measures:

- Syringe calibration
- Instrument preparation
- Instrument zero, span and calibration
- Quality control sample analysis

Instrument Preparation Procedure

1. Freshly prepared drying tubes are required for proper instrument preparation. To prepare the tubes, tightly pack one end of each tube with glass wool. (Wear hand

guards during this operation.) An average packing thickness of one cm should be enough to support the anhydrous magnesium perchlorate (anhydrone). It is important that the glass wool be tightly packed, but it is also important that air can flow through the tube.

2. After all of the tubes have been corked, anhydrone should be carefully poured into the tubes. Granular anhydrone is the best substance to use. Powdered anhydrone can be used in emergencies. Six cm average tube packing should be sufficient. Do not pack the anhydrone into the tube, but allow it to flow in, and do not shake the tube during the process in an effort to pack the anhydrone. When completed, stuff another piece of glass wool into the tube end. After the tubes have been packed, they should be returned to the glass jar to protect them from atmospheric moisture. Tubes can be prepared in advance and stored in an oven (not over 80°C).

3. Insert the freshly prepared drying tubes filled with anhydrous magnesium perchlorate into the instrument.

4. Turn on and warm up the analyzer and recorder for at least 4 hours. It is preferable that the analyzer's main power switch be turned on the night before it is to be used. This will allow the analyzer to achieve thermal equilibrium and for background interference from the electronics to be at a minimum. Check that the compressed air and nitrogen tanks are attached to the proper connections on the Gas Carrier System.

Instrument Zero, Span And Calibration Procedure

Zero

1. After the analyzer has warmed up for at least four hours, ad[illegible]t the gas regulators to 20 psi (nitrogen and air).

2. Place the injection ampule (acid pool reactor) containing 3 mL working acid solution in the instrument assembly and check for a snug fitting around the rubber gas seals.

3. Place two-way valve in "out-to-analyze" position (IR by "Out" pass).

4. Open the ZERO gas toggle valve.

5. Open the FLOW toggle valve.

6. Adjust the IR rate valve to give a rotameter reading of 12 (N_2).

7. Allow the system to flush.

8. Set the instrument meter at zero with the zero control and lock it into position.

9. Set recorder pen to zero and adjust until integrator is lined out (not counting).

Span

10. Set the instrument span to 800.

11. Recheck the zero at the lowest attenuation (one), and if necessary, adjust the base line to zero (leaving the recorder at an attenuation of one).

12. Inject 200 µL of standard (100 mg/L carbonate solution prepared as described in Section 10). The resulting peak height should be 60 ±5% of full scale. If not, repeat the procedure. (If it is not possible to obtain a satisfactory response, refer to the instrument manual for the optical balance adjustment procedure.)

13. Inject 200 µL of standard carbonate solution several more times. Adjust the zero control on the integrator between injections until the response stabilizes and the integrator stops counting when the pen returns to zero.

Instrument Calibration

14. Analyze the standards according to the procedure outlined under analyses of standards and samples in Section 13.

15. Prepare a calibration curve based on the results of the standards analysis. The curve is a plot of carbonate concentration versus instrument response. The calibration curve is generated using response data from analysis of the blanks and calibration standards. The method of least squares fit of data is used to calculate the slope, y-intercept and correlation coefficient of the calibration curve. The validity of the resulting curve is assessed by examining these three items. The slope of the curve is related to sensitivity. Any marked deviation from the average slope for the method indicates that a change in sensitivity has occurred. The y-intercept of the curve should ideally be equal to zero, i.e., instrument response

to a blank should be zero and the calibration curve should pass through the origin. In practice, the curve rarely passes exactly through the origin. The correlation coefficient indicates the amount of scatter of points around the curve. The instrument response should be directly proportional to the carbonate concentration. Perfect correlation of the x and y data points is indicated by a correlation coefficient of 1.0. An acceptable calibration curve should have a correlation coefficient of no less than 0.9950. A recalibration is required for correlation coefficients less than 0.9950.

Syringe Operation And Calibration Procedure

The first step in calibrating a high precision syringe is to become familiar with proper care and filling procedures. The following steps outline these procedures.

1. Rinse the exterior of the syringe with deionized water and dry it completely.

2. Check the glass casing for cracks and chips. If the casing is cracked, it must be replaced.

3. Make sure that the needle is not blocked.

4. Check the adjustment lock ring. It should be finger tight only.

5. Set the syringe at maximum volume, draw deionized water in slowly and eject it slowly. This should be done 7 to 10 times.

6. After rinsing the syringe with deionized water the sample or standard is slowly drawn in by pulling the plunger back to its maximum distance, discarding the liquid and repeating this step 2 times.

7. The aliquot of sample or standard to be analyzed is slowly drawn with the syringe taking care that no bubbles appear in the liquid.

8. Wipe the needle with a clean dry tissue before injecting the solution to be analyzed into an ampule.

The high precision syringe should be calibrated weekly at the 25, 50, 100 and 200 µL settings using deionized water whose density and temperature are recorded. The specified volumes are injected into a standardization vial and the amount injected is weighed using an analytical balance. Determine the average weight of 5 to 7 injections at each position of the syringe (to the nearest 0.0001 g). Calculate the actual volume of the syringe at each setting using the equation:

$$V_{act} = \frac{W_{avg}}{D_t}$$

where: V_{act} = actual volume of syringe (mL),

W_{avg} = average weight of the water injected (g), and

D_t = density of water at the temperature recorded (g/mL).

The data recording form shown in Figure 1 can be used for syringe calibration.

Quality Control Samples

Quality control samples should be analyzed with the frequencies indicated below.

Analyst: ______________________ **Water Temperature:** $t = 24°C$

Date: ______________________ D_t **= Density of Water at** $t = 0.9973$ **g/mL**

Replicate	Weight of Water (g) Contained in Syringe at Setting 25 µL	50 µL	100 µL	200 µL
1	.0287	.0531	.1008	.1986
2	.0283	.0528	.1019	.2002
3	.0285	.0535	.1020	.1993
4	.0288	.0529	.1015	.1989
5	.0281	.0530	.1016	.1996
6	.0284	.0532	.1013	.1995
7	.0285	.0531	.1017	.1995
______				
W_{avg} (g)	.0285	.0531	.1015	.1993
$\frac{W_{avg}}{D_t} = V_{actual}$ (mL)	.0284	.0530	.1012	.1988

Figure 1. Syringe Calibration Data Recording Form with Example Data.

No. of Samples	No. of Full Sets of Standards	No. of Mid-Range Standards	No. of Duplicate Analyses	No. of Blanks	No. of QC Standards	No. of Sample Spikes
1-10	2	1	2	1	1	1
11-20	3	2	3	1	1	2
21-40	3	2	4	2	2	3
40+	3	<10%	<10%	2	2	3

12.0 SAMPLE ACQUISITION AND HANDLING

Samples collected from an FGD system can be unstable (supersaturated) and can contain volatile compounds (carbonate and sulfite). To stabilize the sample, the liquid and solid phases should be separated with a filtration apparatus. The liquid portion should be properly preserved while the solid portion should be dissolved. Procedures for preserving liquid samples and dissolving/preserving solid samples for analysis are outlined below.

Solid Sample Preparation Procedure

1. Wash scrubber solids collected on filter with a 50% acetone/water mixture and dry in a 50-60°C oven. Limestone samples also should be dried, and a higher temperature (90°C) can be used to speed drying because there is no danger of loss of waters of hydration as there is with scrubber solids.

2. Prepare 40 mL disposable septum sample bottles by affixing labels large enough to contain a sample identification and 3 weight figures. Weigh the dry clean bottles to the nearest 0.01 g and record this as "bottle weight" on the label (W_1) and the data recording form.

3. Weigh an amount of solid sample sufficient to contain 2-10 mg CO_3 to the nearest 0.0001 g and transfer the sample to the sample bottle, recording the weight as "sample weight" (SW) in milligrams.

4. Add 25 mL CO_2-free deionized water (prepared by boiling for 10-15 minutes and sealing with an ascarite absorption tube to ventilate while cooling), and tightly seal the sample bottle.

5. Using a 25 mL syringe, evacuate air from the bottle through the septum.

6. Inject 2-3 mL of 1 N HCl into the sample bottle. Allow one hour for the sample to digest and dissolve. Shake the sample occasionally. [Or use a shaker table or other device to agitate samples in large numbers or place sample bottles (especially if they contain coarse or lumpy solids) in an ultrasonic bath until the solids dissolve or disperse finely.] Remove and cool the sample bottles.

7. Inject 3-5 mL of fresh buffer preservative solution into each sample bottle. Invert and shake several times over a period of 1 hour.

8. Using a 25 mL syringe, fill each bottle to the shoulder of the sample bottle with CO_2-free deionized water. (This step is optional.)

9. Shake the sample bottle thoroughly, weigh it to the nearest 0.01 g, and record this weight as the weight of sample bottle plus sample (W_2).

10. Allow all undissolved solids to settle before removing the sample aliquots with a µL syringe. This will prevent damage to the teflon plunger or cylinder walls of the syringe.

11. Determine the specific gravity, S_3, of the solution containing the dissolved solid sample using the method given in this handbook and record (Figure 2a).

12. Use the worksheet in Figure 2 to calculate the dissolution/dilution factor, DF, according to the following equation:

$$DF = \frac{W_2 - W_1}{SW \times S_3}$$

where: DF = dilution/dissolution factor for dissolved solid samples; mL solution/mg sample,

W_2 = weight of sample bottle plus dissolved solid sample from step 9(g),

W_1 = weight of sample bottle from step 2(g),

SW = solid sample weight from step 3 (g), and

S_3 = specific gravity of dissolved solid solution from step 11.

Liquid Sample Preparation Procedure

1. Determine the specific gravity of the liquor sample and the buffer preservative solution using the method described in this handbook. Record these values on the dilution factor worksheet (Figure 2b).

2. Label a clean and dry sample bottle (30 or 50 mL polyethylene or polypropylene bottle), cap the bottle, and weigh to the nearest 0.01 g. Record the weight (W_1) on the sample label and on the data recording form (Figure 2).

Analyst: ____________________

Date: ____________________

S_3 = Specific gravity of dissolved solid sample _____ g/mL

Sample ID	W_2 Weight of Sample Bottle plus Dissolved Sample (g)	W_1 Weight of Sample Bottle (g)	SW Weight of Solid Sample (mg)	$DF = \frac{W_2 - W_1}{S_3 \cdot SW}$

Figure 2a. Worksheet for Calculating Dissolution/Dilution Factor for Solid Samples.

Analyst: ____________________

Date: ____________________

S_2 = Specific gravity of liquor sample ______ g/mL

S_1 = Specific gravity of buffer preservative ______ g/mL

Sample ID	W_1 Weight of Sample Bottle (g)	W_2 Weight of Sample Bottle plus Preservative (g)	W_3 Weight of Bottle, Preservative and Sample (g)	$A = \frac{W_3 - W_2}{S_2}$	$B = \frac{W_2 - W_1}{S_1}$	$DF = \frac{A - B}{A}$
001						

Figure 2b. Worksheet for Calculating Dilution Factor for Liquor Samples.

3. Accurately pipet 5.00 mL of buffer solution into the sample bottle and cap the bottle immediately.

4. Weigh the contents and the bottle to the nearest 0.01 g and record the weight (W_2) on the sample label and the data recording form.

5. Fill the sample bottle to the top with sample, but do not overflow. Cap the bottle and mix thoroughly.

6. Weigh the contents and the bottle to the nearest 0.01 g and record the weight (W_3) on the sample label and on the data recording form.

7. Use the worksheet in Figure 2 to calculate a dilution factor for preserved liquid samples. This factor is used to correct the analytical results. It is calculated using the following equation:

$$DF = \frac{\dfrac{W_3 - W_2}{S_2} + \dfrac{W_2 - W_1}{S_1}}{\dfrac{W_3 - W_2}{S_2}}$$

where: DF = dilution factor (dimensionless),

W_1 = weight of empty sample bottle (g),

W_2 = weight of sample container and preservation solution (g),

W_3 = weight of sample container, buffer preservation solution, and sample (g),

S_1 = specific gravity of buffer preservation solution (g/mL), and

S_2 = specific gravity of the sample (g/mL).

8. Prepare a reagent blank as outlined in steps 1-6 except substitute deionized water for the sample.

13.0 STEPWISE PROCEDURE

Standard and Sample Analysis

1. Make zero and span adjustments according to the procedure outlined in Section 11.
2. Place the two-way valve in the "out-to-analyze" position.
3. Place or replace the injection ampule containing 3 mL working acid solution in the ampule-breaking assembly.
4. Lower the flushing tube well below the acid level.
5. Purge the system with zero gas (N_2) until the instrument has returned to zero.
6. Raise the flushing tube above the acid level.
7. Inject the standard into the acid pool.
8. Lower the flushing tube below the acid level.
9. Check for a flow rate of 12 (N_2).
10. The recorder should show the peak height and integration. Label the peak and record the integration count.
11. Repeat steps 6-10 with: blank and 25, 50, 100, and 200 µL of 100 mg/L carbonate standard.
12. Prepare a calibration curve as described in Section 11 Step 15 and Section 15, Steps 1 and 2.

13. Once an acceptable calibration curve has been prepared, repeat steps 6-10 with the samples. The sample volume injected should give a count near the middle or upper portion of the calibration curve. If it does not, repeat the injection with a smaller volume of sample.

14. Inject standards, QC samples, spikes, and duplicates between samples with the frequency called for in Section 11 (Quality Control).

15. After all samples have been analyzed, turn off all equipment. If samples are to be analyzed the next day, leave the main power source on.

14.0 DATA RECORDING FORM

Figures 1, 2a, 2b, 3 and 4 present example data recording forms.

15.0 CALCULATIONS

1. Prepare a calibration curve showing the relationship between instrument response (peak area) and carbonate concentration in the standards. First, calculate the amount of carbonate, C_{INJ}, in each injection of standard carbonate solution:

$$C_{INJ} = C_{STD} \times V_{ACT}$$

where: C_{INJ} = weight of carbonate injected, µg;
C_{STD} = concentration of carbonate in standard solution, µg/mL;
V_{ACT} = corrected volume of injected solution at syringe setting (from Figure 1), mL.

2. Record the weight of carbonate in each standard injected in an instrument calibration data table like the one shown

Analyst: ____________________ Date Syringe Calibrated ____________

Date Standards Analyzed: ____________________

Standard No.	Syringe Setting (uL)	V_{ACT} (mL)	C_{STD} (ug/mL)	C_{INJ} µg Carbonate Injected	Peak Area (integrator counts)
1	100	0.1028	50	5.14	5000.
2	200	0.2015	50	10.08	10000
Blank	150		0	0	100

m (slope of calibration line) ________

b (y-intercept of calibration line) ________

C_{INJ} = (peak area) - b/m

Figure 3. Instrument Calibration Data Recording Form with Example Data.

Analyst: ____________________

Date: ____________________

Sample ID	Syringe Setting	V_{ACT} mL (Fig. 1)	Peak Area (Integrator Counts)	C_{INJ} µg Carbonate (Fig. 3)	DF Dilution Factor (Fig. 2a)	C µg/mL or µg/mg

$$C = \frac{C_{INJ}}{V_{ACT}} \times DF$$

Figure 4. Data Recording Form for Calculating Carbonate Concentration.

in Figure 3. Also record the corresponding number of integrator counts or peak area for each injection of standard.

3. Use the data in Figure 3 to describe the linear relationship between peak area (Y) and the mg carbonate injected (X). Either plot the data on linear graph paper and use a straightedge to draw the straight line that comes closest to all the data points, or calculate the slope (m) and y-intercept (b) of the line using the method of least squares (Refer to volume I of handbook).

4. Determine the weight of carbonate injected in a sample from the sample peak area (number of integrator counts) and the calibration data (Figure 3). If the calibration line was plotted on a graph with µg carbonate on the x-axis and integrator counts on the y-axis, use the graph to find the weight of carbonate in the sample. Find the number of integration counts for the sample on the y-axis, move horizontally until the calibration line is intersected, then move vertically to find the corresponding value of µg carbonate on the x-axis. Or, if the method of least squares was used, calculate the weight of carbonate injected in a sample from the slope (m) and intercept (b) of the calibration line:

$$C_{INJ} = \frac{\text{(No. of integrator counts)} - b}{m}$$

Record the Sample ID, syringe setting, weight of carbonate injected, C_{INJ}, and the number of integrator counts on the data recording form (Figure 4 for liquids or for solids).

5. Obtain the actual volume of sample injected (V_{ACT}) for the syringe setting from Figure 1 and the dilution factor (DF) for the sample ID from Figure 2a or 2b and record the values on the data recording form (Figure 4).

6. Calculate carbonate concentration in the original (undiluted) liquor or solid sample with the following equation:

$$C_L = \frac{C_{INJ}}{V_{ACT}} \times DF$$

where: C_L = carbonate concentration in solid sample (µg/mg or mg/g), or liquor sample (µg/mL or mg/L),

C_{INJ} = weight of carbonate in injected sample (µg),

V_{ACT} = volume injected at syringe setting (mL), and

DF = dilution factor from Figure 2a or 2b, in mL/mg for solids or dimensionless for liquid samples.

7. Use the following conversion factors to express results in terms of calcium carbonate or carbon dioxide instead of carbonate.

mg/L carbonate x 0.73 = mg/L carbon dioxide
x 1.67 = mg/L calcium carbonate

mg/g carbonate ÷ 10 = wt% carbonate
x 0.73 = mg/g carbon dioxide
x 1.67 = mg/g calcium carbonate

16.0 PRECISION AND BIAS

The precision and bias of carbonate analysis by NDIR absorption spectrophotometry have been investigated (Refer to volume I of handbook). The procedure given in Sections 12 and 13 was used to analyze five synthetic scrubber liquors, four limestones, and two sludge samples from lime FGD systems. The linear least squares curve fit technique was used to prepare calibration curves. Solid samples were digested for one hour.

Table 2 gives the results of determination of single operator precision and bias for carbonate analyses. The results of analysis are expressed as mg carbonate per liter for synthetic liquors and mg carbonate per gram for limestone and sludges.

The pooled coefficient of variation for synthetic liquor samples containing calcium, magnesium, sodium, sulfate, chloride, and 6 to 36,000 mg/L carbonate was 4.0 percent. The range of bias based on known amounts of carbonate added was -16 to 39 percent, although significant bias was not found for the sodium carbonate sample or the magnesia scrubbing liquor.

The pooled coefficient of variation for one reagent grade calcium carbonate sample and four limestone samples was 5.3 percent; percent bias ranged from -6. to 3.9 percent. No significant bias was found for any of the four limestones. The pooled coefficient of variation for solid samples from lime FGD systems was 8.4 percent.

Table 3 summarizes the precision limits for carbonate analysis on the three materials tested. The single-operator coefficient of variation (one sigma limit) and the difference two sigma limit are given. The results of two properly conducted analyses by the same analyst on the same material using the same instrument should not differ by more than the percent of their average shown in the last column of Table 3.

17.0 REFERENCES

1. J.W. Molina. *Aqueous CO_2 - Oceanography CO_2 Analyzer*. Radian Corporation Technical Note 200-403-50, 1973.

Table 2

RESULTS OF INVESTIGATION OF PRECISION AND BIAS OF CARBONATE ANALYSIS IN SCRUBBER LIQUOR, SOLIDS AND LIMESTONE BY NDIR SPECTROPHOTOMETRY

Sample ID and Description[1]	True Value[2] (mg/L or mg/g)	n = number of Replicates[3]	Mean of Measurements (mg/L or mg/g)	Precision[4]		Bias[5] (%)
				Standard Deviation (mg/L or mg/g)	Coefficient of Variation (%)	
Synthetic Liquid Samples						
QCCG-Na_2CO_3 in deionized water	100.	6	102.2	4.1	4.1	2.2*
LQ5-High TDS limestone scrubber liquor	6.0	8	8.32	0.5	6.0	39.
LQ1-High magnesium lime scrubber liquor	30.	8	31.	1.1	3.4	3.2*
LQ2-Magnesia (high Mg) scrubber liquor	120.	8	157.	6.6	4.2	31.
LQ4-Low TDS limestone scrubber liquor	300.	8	262.	5.5	2.1	-13.
LQ3-Dual alkali scrubber liquor	36,000.	8	30,300.	1,040.	3.4	-16.
Solid Samples						
Reagent grade calcium carbonate	600.	6	575.	35.3	6.1	-4.1*
LS-1 Monteagle limestone	550.	6	550.	44.	7.9	0*
LS-2 Ciloo limestone	572.	5	591.	17.	2.9	3.3*
LS-3 Brassfield limestone	576.	7	599.	23.	3.8	3.9*
LS-4 NBS dolomite	636.	7	595.	30.	5.0	-6.4*
SL1-Lime spray dryer product (80% fly ash)		8	12.0	0.7	6.0	NA
SL1-Lime scrubber solids		8	15.4	1.6	10.3	NA

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[1] Sample ID

Sample ID	Composition (mg/L)					
	Ca	Mg	Na	SO_4	CO_3	Cl
LQ1	400	1,215	379	6,144	30	284
LQ2	160	36,500	1,200	146,000	120	285
LQ3	40	122	45,800	48,000	36,000	14,300
LQ4	561	243	643	2,570	300	600
LQ5	20,000	608	237	768	6	35,800

[2] For synthetic liquors, the true value is the known amount of reagent grade chemicals added to deionized water. For limestone samples the true value is based on repeated measurements using a number of different analytical methods.

[3] Number of times a separate sample of the same material was analyzed by the same analyst with the same instrument and a separate calibration

[4] Standard deviation = $[\Sigma(X_i - \bar{X})^2/n-1]^{1/2}$

Coefficient of variation = (standard deviation/mean) x 100

[5] Bias = [(mean - true value)/true value] x 100

*Not statistically significant at the 0.95 level of confidence

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Table 3

PRECISION LIMITS FOR ANALYSIS OF CARBONATE IN LIMESTONE AND SCRUBBER LIQUORS AND SOLIDS BY NDIR ABSORPTION SPECTROPHOTOMETRY

Material	Single Operator Coefficient of Variation* (1S%)	Difference Two Sigma Limit* (D2S%)
Synthetic scrubber liquors containing 6 to 36,000 mg/L carbonate as well as Ca, Mg, Na, SO_4 and Cl	4.0	11.3
Limestone	5.3	15.0
Solids from lime FGD systems	8.4	23.8

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*These numbers represent, respectively, the (1S%) and (D2S%) limits described in ASTM Practice C670-81 for preparing precision statements for test methods for construction materials.

2. S. A. Schubert and K. L. Kelly. *Demonstration of Adipic Acid Addition on a Full-Scale Operating Limestone FGD System--Analytical Procedures Manual for City Utilities of Springfield Southwest Power Plant*. Radian Corporation DCN 80-213-012-11, prepared for the U.S. EPA, November 1980.

3. K. Schwitzgebel, et. al. *Development of Sampling and Analytical Methods of Lime/Limestone Wet Scrubbing Tests*. Vol. I&II. Final Report OAP Contract No. CPA TO-143, Radian Project No. 200-006, 1971.

4. B. F. Jones, et. al. *Experimental and Theoretical Studies of Solid Solution Formation in Lime and Limestone SO_2 Scrubbers*. Vol. II, Appendices. Radian Corporation, EPA 600/2-76-2736, October 1976.

5. *Laboratory Procedures Manual*. Shawnee Test Facility. Prepared by Bechtel National, March 1976.

6. C. Galloway and P. Nassos. Radian Laboratory Notebook No. 00645. October 1982.

APPENDIX D

PRETEST WASTE CHARACTERIZATION ANALYTICAL RESULTS

Date: December 24, 1987

To : Sy Rosenthal

From: Renee G. Cohen

Thru: James Chang JC
Marion Murphey MM
Janis Giga

On August 28, 1987 one (1) 16 oz. solid sample identified as Shirco Rose Composite was sent to Radian Laboratories Inc. for the following analyses:dioxin, polychlorinated biphenyls, and lead. There was to be one matrix spike/ matrix spike duplicate (MS/MSD) performed with each analysis. The analyses were performed according to the following publication:

> EPA Test Methods for the Evaluation of Solid Waste.
> Third Edition. September 1986.

Enclosed please find the case narrative submitted by Nathan Cobb for the lead and PCB analysis. The summary tables and QA/QC data is found following this memo. The dioxin data was submitted at a later date and is found preceding this memo as well. All the supporting data, laboratory correspondence and chain of custodies have been included for your information.

CASE NARRATIVE

Soil sample P7-09-006-01a was received from the field sampling 8-28-87 for analysis. The sample was to be analyzed for lead, PCB's, and dioxins. Aliquots were also to be taken and spiked as a matrix spike and a matrix spike duplicate. A Method Blank was to be run for each analysis.

The sample was aliquoted for PCB analysis and prepared by Method 3550 Sonication Extraction for analysis by Method 8080 GC analysis for PCB's. Aliquots were also taken for lead analysis and prepared by Method 3050 and analyzed by Method 7420. The sample was aliquoted for preparation and analysis of Dioxins by Method 8280.

The PCB analysis identified 140 ug/g of Aroclor 1260 and 486 ug/g of Aroclor 1248 in the soil sample. The Method Blank was free of contaminants and 74% of the DBC internal spike was recovered. The Matrix Spikes were masked by the Aroclors in the sample.

The lead analysis showed 564 ug/g of lead in the soil. The Method Blank was uncontaminated and the Matrix Spikes were recovered at 98% and 100%.

The sample was prepared for analysis of dioxins, but at the time of this documentation, the analysis had not yet been completed.

SUMMARY

PCB s

Sample	P7-09-006-01a	140 ug/g Arochlor 1260 486 ug/g Arochlor 1248
	P7-09-006-02a P7-09-006-03a	Not identifyable Reference p. 17
	P7-09-006-04a	No Arochlor identified 74% DBC surrogate recovered Reference p. 14

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TABLE (). RESULTS OF LEAD IN SOIL ANALYSIS

All concentrations reported in ug/g

Sample Identification	Concentration
Shirco Rose Composite	560
Method Blank	ND (1.5)

ND denotes not detected.

QA/QC TABLE (). RESULTS OF MATRIX SPIKE/MATRIX SPIKE DUPLICATE OF LEAD IN SOIL ANALYSIS

All concentrations reported in ug/g

Sample Identification: Shirco Rose Composite

SAMPLE CONC.	SPIKE CONC.		RECOVERED CONC.		PERCENT RECOVERY		RPD
	MS	MSD	MS	MSD	MS	MSD	
560	19000	20000	20000	20000	102	97	5.0

RPD denotes relative percent difference.

C 525

TABLE 1. SOIL DIOXIN/FURAN RESULTS (ng/g)

Analyte	36952 A710002-01A P709004-01A Soil	36965 A710002-04A P709004-04A Reagent Blank
Total TCDD	55	<0.2
Total TCDF	4.2	<0.2
Total PCDD	<0.4	<0.2
Total PCDF	<0.3	<0.2
Total HxCDD	<0.6	<0.3
Total HxCDF	<0.5	<0.2
Total HepCDD	<0.3	<0.4
Total HepCDF	<0.6	<0.3
OCDD	<0.9	<0.9
OCDF	<0.7	<0.7

TABLE 2. SOIL DIOXINS/FURANS SURROGATE RECOVERIES, (%)

Surrogate	36952 A710002-01A P709004-01A Soil	36953 A710002-02A P709004-02A Matrix Spike	36954 A710002-03A P709004-03A Matrix Spike	36965 A710002-04A P709004-04A Blank
13C-2,3,7,8-TCDD	29	26	33	57
13C-2,3,7,8-TCDF	31	23	33	57
13C-1,2,3,7,8-PCDD	27	7	30	56
13C-1,2,3,7,8-PCDF	29	8	30	52
13C-1,2,3,6,7,8-HxCDD	27	6	31	56
13C-1,2,3,4,7,8-HxCDF	25	4	23	54
13C-HepCDD	59	24	37	47
13C-HepCDF	23	4	22	43
13C-OCDD	36	23	53	25
13C-OCDF	26	40	66	25

TABLE 3. SOIL DIOXIN/FURAN MATRIX SPIKE RESULTS

Compound	Initial Conc. ng/g	Amount Added ng/g		Amount Recovered ng/g		% Recovery	
		1st	2nd	1st	2nd	1st	2nd
2,3,7,8-TCDD	<0.4	9.5	9.3	7.8	8.3	82	90
1,2,3,7,8-PCDD	<0.4	9.5	9.3	8.7	7.0	91	76
1,2,3,4,7,8-HXCDF	<0.5	9.5	9.3	10	12	110	120
1,2,3,4,6,7,8-HpCDD	<0.3	9.5	9.3	5.1	6.3	54	68
OCDD	<0.9	22	22	31	17	140	81

ENVIRESPONSE, INC.
ENVIRONMENTAL EMERGENCY RESPONSE UNIT
GSA RARITAN DEPOT, WOODBRIDGE AVENUE, BUILDING 209, BAY F, EDISON, N.J. 08837
(201) 548-9660

DATE: March 15, 1988

TO: Sy Rosenthal

FROM: Renee G. Cohen

THRU: Janis Giga Jan Giga

This memo is the compilation of all data from the initial sampling of the Shirco Rose Site in Rose Township, Michigan. The laboratory subcontracted to complete the analyses requested was Analytikem Laboratories in Cherry Hill, NJ. Enclosed find all the tabulated results, raw data as well as any sample chromatograms or instrument printouts. Any QA/QC work completed by the outside laboratory is included and the discussion of this data is presented in Section II.

Enclosure

tm/Rosenthal/M-3-15

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Introduction

On July 16, 1987 Enviresponse, Inc. received thirty seven (37) soil samples from the Shirco site in Rose Township, Michigan. These samples were sent to AnalytiKEM Laboratories on July 20, 1987. The analyses requested for these samples included polychlorinated biphenyls (PCBs) and lead analysis. Sample A06577 was a physical composite. This sample was analyzed for PCBs and lead plus elemental analysis, pH, percent moisture, percent ash, BTU and density.

ANALYTICAL PROCEDURES

PCB Analysis:

The PCBs were analyzed by Method 608 modified. The soil samples were soxhlet extracted with methylene chloride, then solvent exchanged to hexane subjected to a florisil cleanup, and concentrated to a final volume of 10 mls.

The samples contained varying amounts of Aroclors 1248 and 1254. The fingerprints for the standards were not as well resolved as one would expect. There were Aroclor mixtures in almost all of the samples, and because the standards had poor resolution it was difficult to accurately quantify each Aroclor present. The analysis, however, is accurate enough for the the purpose intended.

The results of the analysis are presented in Table 1. Please note the values in parentheses are the detection limits.

Lead Analysis:

The samples were digested and analyzed according to the following publication:

EPA Test Methods for Evaluating Solid Waste, 1982.

The digestion procedure (Method 3050) was the one used for soils and analyzed by graphite furnace (Method 7421). These samples were analyzed by inductively coupled plasma (ICP). In the case of lead the variation in the digestion method will not affect the concentration of the lead in the sample. Table 2 presents the results of lead in soil analysis.

The results of the physical testing are presented in Table 3. The analyses were performed according to the following methods:

Standard Methods, 15th Edition for pH

ASTM Methods, D240, D95, D482 for heat, ash, moisture

EPA Test Methods for Evaluating Solid Waste, 1982 for density

Table 4 presents the results of the elemental analysis. These analyses were performed according to the various ASTM methods.

Section IV of this report contains a brief description of the methods used in the above analysis.

TABLE 1. RESULTS OF PCBs IN SOIL ANALYSIS

All concentrations reported in ug/g

Constituent	S1 A06447	S2 A06416	S3 A06418	S4 A06419
Aroclor 1016	ND (.33)	ND (3.33)	ND (3.3)	ND (33)
Aroclor 1221	ND (.33)	ND (3.33)	ND (3.3)	ND (33)
Aroclor 1232	ND (.33)	ND (3.33)	ND (3.3)	ND (33)
Aroclor 1242	ND (.33)	ND (3.33)	ND (3.3)	ND (33)
Aroclor 1248	18	31	100	940
Aroclor 1254	52	18	69	330
Aroclor 1260	ND (.33)	ND (3.3)	ND (3.3)	ND (33)

ND denotes not detected. The value in parentheses is the method detection limit.

TABLE 1. RESULTS OF PCBs IN SOIL ANALYSIS (CONT'D)

All concentrations reported in ug/g

Constituent	S5 A06417	S6 A06435	S7 A06436	S8 A06441
Aroclor 1016	ND (.33)	ND (.33)	ND (33)	ND (3.3)
Aroclor 1221	ND (.33)	ND (.33)	ND (33)	ND (3.3)
Aroclor 1232	ND (.33)	ND (.33)	ND (33)	ND (3.3)
Aroclor 1242	ND (.33)	ND (.33)	ND (33)	ND (3.3)
Aroclor 1248	5.4	14	85	46
Aroclor 1254	24	14	47	25
Aroclor 1260	ND (.33)	ND (.33)	ND (33)	ND (3.3)

ND denotes not detected. The value in parentheses is the method detection limit.

TABLE 1. RESULTS OF PCBs IN SOIL ANALYSIS (CONT'D)

All concentrations reported in ug/g

Constituent	S9 A06437	S10 A06442	S11 A06438	S12 A06444	S13 A06439
Aroclor 1016	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1221	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1232	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1242	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1248	150	900	70	ND (3.3)	140
Aroclor 1254	82	330	110	29	60
Aroclor 1260	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)

ND denotes not detected. The value in parentheses is the method detection limit.

TABLE 1. RESULTS OF PCBs IN SOIL ANALYSIS (CONT'D)

All concentrations reported in ug/g

Constituent	S14 A06443	S15 A06440	S16 A06445	S17 A06446	S18 A06448
Aroclor 1016	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1221	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1232	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1242	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1248	110	440	210	260	71
Aroclor 1254	54	170	91	100	44
Aroclor 1260	ND (3.3)	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)

ND denotes not detected. The value in parentheses is the method detection limit.

TABLE 1. RESULTS OF PCBs IN SOIL ANALYSIS (CONT'D)

All concentrations reported in ug/g

Constituent	S19 A06450	S20 A06449	S21 A06693	S22 A06559
Aroclor 1016	ND (3.3)	ND (3.3)	ND (33)	ND (33)
Aroclor 1221	ND (3.3)	ND (3.3)	ND (33)	ND (33)
Aroclor 1232	ND (3.3)	ND (3.3)	ND (33)	ND (33)
Aroclor 1242	ND (3.3)	ND (3.3)	ND (33)	ND (33)
Aroclor 1248	38	24	1,100	860
Aroclor 1254	28	23	450	440
Aroclor 1260	ND (3.3)	ND (3.3)	ND (33)	ND (33)

ND denotes not detected. The value in parentheses is the method detection limit.

TABLE 1. RESULTS OF PCBs IN SOIL ANALYSIS (CONT'D)

All concentrations reported in ug/g

Constituent	S23 A06694	S24 A06560	S25 A06695	S26 A06698
Aroclor 1016	ND (33)	ND (33)	ND (33)	ND (3.3)
Aroclor 1221	ND (33)	ND (33)	ND (33)	ND (3.3)
Aroclor 1232	ND (33)	ND (33)	ND (33)	ND (3.3)
Aroclor 1242	ND (33)	ND (33)	ND (33)	ND (3.3)
Aroclor 1248	1,000	800	450	89
Aroclor 1254	410	320	100	39
Aroclor 1260	ND (33)	ND (33)	ND (33)	ND (3.3)

ND denotes not detected. The value in parentheses is the method detection limit.

TABLE 1. RESULTS OF PCBs IN SOIL ANALYSIS (CONT'D)

All concentrations reported in ug/g

	S27	S28	S29	S30
Constituent	A06696	A06699	A06697	A06700
Aroclor 1016	ND (33)	ND (33)	ND (3.3)	ND (33)
Aroclor 1221	ND (33)	ND (33)	ND (3.3)	ND (33)
Aroclor 1232	ND (33)	ND (33)	ND (3.3)	ND (33)
Aroclor 1242	ND (33)	ND (33)	ND (3.3)	ND (33)
Aroclor 1248	190	190	46	180
Aroclor 1254	80	72	ND (3.3)	110
Aroclor 1260	ND (33)	ND (33)	ND (3.3)	ND (33)

ND denotes not detected. The value in parentheses is the method detection limit.

TABLE 1. RESULTS OF PCBs IN SOIL ANALYSIS (CONT'D)

All concentrations reported in ug/g

Constituent	S31 A06561	S32 A06573	S33 A06562	S34 A06574
Aroclor 1016	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1221	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1232	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1242	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)
Aroclor 1248	700	200	15	82
Aroclor 1254	320	95	13	66
Aroclor 1260	ND (33)	ND (3.3)	ND (3.3)	ND (3.3)

ND denotes not detected. The value in parentheses is the method detection limit.

TABLE 1. RESULTS OF PCBs IN SOIL ANALYSIS (CONT'D)

All concentrations reported in ug/g

Constituent	S35 A06563	S36 A06575	Physical Composite A06577
Aroclor 1016	ND (3.3)	ND (33)	ND (33)
Aroclor 1221	ND (3.3)	ND (33)	ND (33)
Aroclor 1232	ND (3.3)	ND (33)	ND (33)
Aroclor 1242	ND (3.3)	ND (33)	ND (33)
Aroclor 1248	70	430	420
Aroclor 1254	33	130	150
Aroclor 1260	ND (3.3)	ND (33)	ND (33)

ND denotes not detected. The value in parentheses is the method detection limit.

rhg/ana-shirco

TABLE 2. RESULTS OF LEAD IN SOIL ANALYSIS

Concentrations reported in ug/g (ppm)

Sample ID	Location	Concentration
Method Blank		ND (10)
A06447	S1	100
A06416	S2	240
A06418	S3	310
A06419	S4	370
A06417	S5	740
A06435	S6	560
A06436	S7	400
A06441	S8	130
A06437	S9	240
A06442	S10	330
A06438	S11	530
A06444	S12	1,100
A06439	S13	450
A06443	S14	400
A06440	S15	220
A06445	S16	210
A06446	S17	340
A06448	S18	140
A06450	S19	680
A06449	S20	440
A06693	S21	240
A06559	S22	160
A06694	S23	200
A06560	S24	180
A06695	S25	300
A06698	S26	240
A06696	S27	390
A06699	S28	93
A06697	S29	260
A06700	S30	180
A06561	S31	460
A06573	S32	380
A06562	S33	ND (10)
A06574	S34	81
A06563	S35	110
A06575	S36	340
A06577	Physical Composite	580

ND denotes not detected. Value in parentheses is the method detection limit.

rhg/ana-shirco

TABLE 3. RESULTS OF PHYSICAL TESTING

Sample No: A06577 - Physical Composite

Parameter	Concentration #
pH	7.8
Moisture	9%
Ash	82%
Heat of Combustion	*
Density	0.95 g/ml

* denotes the sample did not combust
# denotes the average of duplicate analyses

rhg/ana-shirco

TABLE 4. RESULTS OF ELEMENTAL ANALYSES

Sample A06577 - Physical Composite

Parameter	Concentration *
Sulfur	321 ppm
Phosphorus	0.042 %
Chlorine	0.15 %
Carbon	2.27 %
Hydrogen	0.84 %
Nitrogen	ND (0.06) %
Oxygen	4.06 %

ND denotes not detected. The value in parentheses is the detection limit.
* denotes the average of duplicate analysis.

QA/QC PROCEDURES

PCB Analysis

QA/QC Table 5 presents the results of the surrogate standard recoveries. Most of the samples had no recovery reported for the surrogate because the samples had such large dilution factors.

QA/QC Table 6 presents the results of the matrix spike duplicate recoveries. The spike recoveries ranged from 12-50%. These recoveries are low, but were within the AnalytiKEM laboratory acceptable limits.

Lead Analysis

QA/QC Table 7 presents the results of the matrix spike/matrix spike duplicate recoveries for the lead analysis. All the samples listed in QA/QC Table 7 were spiked with 1000 ug of lead. The spike concentrations in ug/g in the table varied due to the weight of the sample digested in the spike and spike duplicate. The sample was analyzed one time, the difference in the weight between the MS and MSD sample analysis are due to the differences in weights of the digested spike samples. All samples had a spike recovery of lead in both the MS and MSD between 80 and 86 percent. The exception was in sample A06577 which had a spike recovery of 65 and 64 percent. This sample might possibly have matrix interferences which led to the low recovery of the matrix spike and matrix spike duplicate. The relative percent difference (RPD) of all the samples was below 4.7.

QA/QC Table 8 presents the duplicate results of the physical testing parameters. The RPD of the duplicate runs in all cases was less than 1.2 in the cases where an RPD could be calculated. Sample A06577 did not combust in either of the two runs so the RPD of this parameter was not calculated.

QA/QC Table 9 presents the duplicate results of the elemental analysis. The RPD of the parameters analyzed was between 0.0 and 23.8. The RPD of nitrogen was not calculated because in the duplicate runs the concentration of nitrogen in the sample is reported as not detected.

QA/QC TABLE 5. SURROGATE STANDARD RECOVERIES FOR PCBs IN SOIL

Sample ID	Site Location	Percent Recovery
A06447	S1	100
A06416	S2	101
A06418	S3	52
A06419	S4	NR
A06417	S5	80
A06435	S6	NR*
A06436	S7	NR
A06441	S8	92
A06437	S9	NR
A06442	S10	NR
A06438	S11	NR
A06444	S12	NR
A06439	S13	NR
A06443	S14	NR
A06440	S15	NR
A06445	S16	NR
A06446	S17	NR
A06448	S18	NR
A06450	S19	NR
A06449	S20	NR
A06693	S21	NR
A06559	S22	NR
A06694	S23	NR
A06560	S24	NR
A06695	S25	NR
A06698	S26	NR
A06696	S27	NR
A06699	S28	NR
A06697	S29	NR
A06700	S30	NR
A06561	S31	NR
A06573	S32	NR
A06562	S33	NR
A06574	S34	NR
A06563	S35	NR
A06575	S36	NR
A06577	Physical Composite	NR

NR - denotes not recovered due to dilutions.
NR* - denotes not recovered due to matrix interferences.

QA/QC TABLE 5. SURROGATE STANDARD RECOVERIES FOR PCBs IN SOIL (CONT'D)

QA/QC Samples	Percent Recovery
A06419 S4	
MS	NR
MSD	NR
A06440 S15 MS	
MS	NR
MSD	NR
A06445 S16	
MS	NR
MSD	NR
A06698 S26	
MS	NR
MSD	NR
A06577 Physical Composite	
MS	NR
MSD	NR

NR denotes not recovered due to dilutions.
MS denotes matrix spike.
MSD denotes matrix spike duplicate.

QA/QC TABLE 6. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RESULTS FOR POLYCHLORINATED BIPHENYLS IN SOIL

All samples were spiked with 500 ug of Aroclor 1260

Sample ID	Site Location	Sample Conc.	Spiked Conc.	Recovered Conc. MS	Recovered Conc. MSD	Percent Recovery MS	Percent Recovery MSD	RPD
A06419	S4	ND (33)	500	245	60	46	12	117.0
A06440	S15	ND (33)	500	225	225	45	45	0.0
A06445	S16	ND (3.3)	500	215	245	43	49	13.0
A06698	S26	ND (3.3)	500	235	200	47	44	7.0
A06577	Physical Composite	ND (33)	500	250	130	50	26	64.0

RPD denotes relative percent difference.
ND denotes not detected. The value in parentheses is the method detection limit.

QA/QC TABLE 7. MATRIX SPIKE/MATRIX SPIKE DUPLICATE FOR LEAD ANALYSIS

All concentrations reported in ug/g (ppm)

Sample ID	Site Location	Sample Conc.*		Spike Conc.		Recovered Conc.		Percent Recovery		RPD
		MS	MSD	MS	MSD	MS	MSD	MS	MSD	
A06419	S4	370	370	1000	1000	1200	1180	83	81	0.0
A06440	S15	230	250	1000	909	1090	1010	86	84	2.3
A06445	S16	230	250	909	833	970	920	81	80	1.2
A06698	S26	260	240	909	1000	1060	1080	88	84	4.7
A06577	Physical Composite	580	580	1000	1000	1230	1220	65	64	1.6

RPD denotes relative percent difference.
* sample concentrations were adjusted to account for weight differences of samples and spikes.

QA/QC TABLE 8. DUPLICATE RESULTS OF PHYSICAL TESTING

Sample No: A06577 - Physical Composite

Parameter	Sample Conc		RPD
	Run 1	Run 2	
pH	7.8	7.8	0.0
Moisture	9%	9%	0.0
Ash	81%	82%	1.2
Heat of Combustion	*	*	---
Density	0.95 g/ml	0.95 g/ml	0.0

RPD denotes relative percent difference.
* denotes sample and its duplicate did not combust

QA/QC TABLE 9. RESULTS OF DUPLICATE ELEMENTAL ANALYSIS

Sample ID: A06577 - Physical Composite

Parameter	Sample Conc Run 1	Sample Conc Run 2	RPD
Sulfur	308 ppm	333 ppm	7.8
Phosphorus	0.047%	0.037%	23.8
Chlorine	0.15%	0.15%	0.0
Carbon	2.15%	2.38%	10.2
Hydrogen	0.78%	0.90%	14.3
Nitrogen	ND (0.06)%	ND (0.05)%	----
Oxygen	3.92%	4.20%	6.9

RPD denotes relative percent difference.
ND denotes not detected the value in parentheses is the method detection limit.

APPENDIX E

TEST PLAN FOR THE MONITORING OF VAPOR PHASE LEAD EMISSIONS FROM THE SHIRCO PORTABLE PILOT UNIT DURING THE ROSE TOWNSHIP DEMONSTRATION TESTS

274-013-04-01

TEST PLAN FOR THE MONITORING OF VAPOR PHASE LEAD EMISSONS FROM THE SHIRCO PORTABLE PILOT UNIT DURING THE ROSE TOWNSHIP DEMONSTRATION TESTS

Presented to:

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1.0 INTRODUCTION

Radian Corporation under contract to Enviresponse, Inc., will conduct a comprehensive characterization of Shirco Infrared Systems' Portable Pilot Test Unit. The program will be performed under the auspices of EPA's Superfund Innovative Technology Evaluation (SITE) program at the Rose Township site near Flint, Michigan. The site is known to be contaminated with PCBs and lead.

Many of the environmental issues relating to the incineration of hazardous wastes or contaminated materials have revolved around the destruction of organic constituents. However, recently there has been a growing awareness concerning the emission of metals associated with the organically contaminated materials into the atmosphere.

At the Rose Township site, as with many others, metals present a significant environmental concern. The approximately maximum concentration of lead at Rose Township is roughly 50 percent greater than that of PCBs (lead: 1,480 ppm and PCBs: 980 ppm). Experience at another site using Shirco technology (Peak Oil), indicates that lead is vaporized from the soil in the primary furnace and later recondenses as the hot flue gases are cooled forming a very fine particle which is very difficult to remove using conventional wet scrubbers.

Radian Corporation, through work with the Electric Power Research Institute and the Department of Energy, has developed an on-going, continuous vapor phase monitor for selected metals, including lead. This system provides the unique capability of providing "real time" data which can allow a process evaluation for the control of metals emissions. The Rose Township program provides an excellent opportunity to evaluate process performance relative to not only the destruction of organics, but also the control of metals (specifically lead) emissions.

Radian will perform a comprehensive characterization of the incineration process during the initial three-day test. The project then includes a six-day parametric test including 15 operating scenarios. During the parametric tests, a lesser scope of work will be conducted. The results of continuous vapor analysis can be compared to particulate emission data to be generated by Radian during all phases of the Rose Township test to evaluate metals (Pb) emissions relative to particulate emissions.

This test plan presents Radian's approach to the continuous monitoring of vaporous lead in the primary furnace off-gas from the Shirco Portable Pilot Test Unit during the Rose Township demonstration tests. These data can be used to determine the incinerator operating conditions required to destroy the organic material while minimizing the volatilization of lead from the contaminated soil.

2.0 SCOPE OF WORK

Radian will perform this program in four separate tasks:

- Task 1 -- Test Plan Preparation and Method Validation;
- Task 2 -- On-Site Analysis;
- Task 3 -- Off-Site Analysis; and
- Task 4 -- Data Evaluation and Reporting.

2.1 Task 1 -- Test Plan Preparation and Method Validation

Under Task 1 the detailed test plan has been prepared. Also under Task 1, the specific adaptation of Radian's on-line, continuous vapor phase trace element monitor for the primary furnace off-gas has been developed. A vapor phase lead standard was used to define the vapor phase lead to liquid phase lead response factor so that calibration of the system is simplified to aqueous standards for field use. In addition, the sampling location was defined and the sampling method developed. The protocol for on-site data collection and frequency of data collection was defined.

2.1.1 Instrument Check

Following is the description of the steps used to optimize instrumental response for lead in an air-simulated primary off-gas-acetylene flame.

Flame Optimization and Calibration

1. Turn the AA on and insert the Pb hollow cathode lamp. Set the lamp amperage at 5.0 mA and let warn up for about 20 minutes.

2. Make up lead standards from a commercially available stock solution (1,000 mg/L) at 2, 4, 10, 16, 20 mg/L Pb. From a separate stock source make up QC standards of 8 and 14 mg/L Pb.

3. Optimize the wavelength around 283.0 nm and adjust the lamp height and angle.

4. Ignite the air acetylene flange. Optimize air and acetylene flows (approximately 4.5 and 1 SLM, respectively).

5. Adjust the burner head height and orientation until the maximum absorbance for the 10 mg/L standard is obtained. Optimize the nebulization rate.

6. Nebulize the liquid standards and record the response in absorbance. Use this data to determine the linear range and detection limits for this flame gas mixture.

7. Measure the nebulization rate and record.

8. Be sure to record gas flows, lamp amperage, PM Tube voltage, nebulization rate and date.

9. Slowly add in the simulated primary furnace off-gas (blend) to the oxidant while reducing the air flow. Continue this until the maximum amount of blend is added to the flame while maintaining a stable flame across the burner head.

10. Repeat steps 5 through 8.

Nebulization Efficiency

The nebulization efficiency was determined experimentally. The nebulization efficiency is used in calculations to determine vapor pahse concentrations from liquid phase response (see 2.1.2, step 9).

1. Empty the burner-head drain tube.

2. Add a known amount of water (approximately 10 mL) to the drain tube. Reattach this tube to the burner lead assembly (W_1).

3. Weigh the waste collection vessel (W_2). Place the drain tube in this vessel.

4. Weigh a beaker full of water (approximately 400 mL) (W_3).

5. Set up the instrument with the appropriate air-blend-acetylene flame. Nebulize the water from the beaker. Continue for 30-60 minutes.

6. Take the nebulization tube out of the beaker and turn off the flame. Weigh the beaker (W_4).

7. Detach the drain tube and drain the water in it to the waste vessel. Weigh the waste vessel (W5).

8. Nebulization Efficiency (%) = $\dfrac{(W_3 - W_4)}{W_5 - (W_1 + W_2)} \times 100.$

9. Repeat.

2.1.2 Vapor Phase to Liquid Phase Response Factor

A correction factor to be applied to the field data was established by comparing the calculated vapor phase lead concentration determined by the methods of standard additions with liquid standards and the actual vapor phase lead concentration. A vapor phase lead standard, tetraethyl lead, was generated.

1. Prepare a vapor phase lead standard by placing approximately 15 mL of tetraethyl lead into a gas mixing vessel. Insert a small stir bar. Place ball valves on the inlet and outlet ports and close. Allow to stand and equilibrate for 24 hours. (Place vessel in water so that leaks in the plumbing may be detected by watching the water for bubbles).

2. Plumb the vessel with the inlet port to the standard rotameter exit and the vessel exit to the fuel line.

3. Place the vapor phase lead standard vessel in a constant temperature water bath (approximately 28°C) and turn on stir slate.

4. Set up the AA and calibrate with the liquid phase standard in the air-blend-acetylene flame.

5. Open the inlet and outlet ball valves on the vapor phase standard vessel. Using nitrogen as a feed gas to the standard rotameter add a known volume of the vapor phase lead standard to the flame (R=10, R=15).

6. Obtain a steady reading of the standard rotameter setting. Calibrate with liquid phase standards.

7. Repeat steps 5 and 6 at least three times at each standard rotameter setting.

8. Use the absorbance readings for the liquid phase standards and the initial zero for each rotameter setting to perform the method of standard additions and calculate the amount of Pb delivered to the flame (as ug/mL Pb).

9. Convert the result obtained in step 8 to vapor phase concentration using the equation:

$$\frac{X\ \text{ug pB}}{\text{mL Standard}} \times \frac{6.90\ \text{mL Standard}}{\text{minute}} \times \frac{\text{umole Pb}}{207\ \text{ug Pb}} \times \frac{24.463\ \text{ul Pb}}{\text{umole Pb}} \times \frac{\text{minute}}{2.26\ \text{L blend gas}}$$

$$= \frac{\text{ul Pb}}{\text{L blend}}.$$

10. Calculate the vapor pressure of vapor phase lead from the vapor pressure equation on age D-158 of the 54th edition of CRC.

$$\log P_b = (-0.2185\ A/K) + B$$

where A = 12959.7 cal/mole
B = 9.120674
P = pressure in Torr
K = temperature in degrees Kelvin.

11. Use the ideal gas law and the standard flow rate to determine the actual concentration of the vapor phase standard.

$$n = \frac{PV}{RT}$$

where P = pressure of tetraethyl lead in Torr
V = volume of standard vessel in liters.
$R = 62.4 \frac{\text{Torr} \cdot \text{L}}{\text{mole} \cdot \text{K}}$
T = temperature in degrees Kelvin.

Then convert moles of tetraethyl lead to 1 ul Pb/L blend gas using the following relationship:

$$\frac{\text{moles TEPb}}{} \times \frac{\text{mole Pb}}{\text{mole TEPb}} \times \frac{\text{24.463 L Pb}}{\text{mole Pb}} \times \frac{\text{10-6 ul}}{\text{1L}} \times \frac{\text{minute}}{\text{2.26 L blend}} \times \frac{\text{volume Pb standard delivered to flame (L)}}{\text{minute}}$$

$$= \frac{\text{ul Pb}}{\text{L blend}} .$$

12. Establish the relationship between the vapor phase concentration calculated from the liquid phase standards and the actual vapor phase concentration. This relationship will be used to correct the field data which will be obtained from liquid phase standards.

2.1.3 Preparation of Sampling Apparatus

A sample probe for the primary furnace off-gas was constructed and calibrated as described below.

1. Obtain a ceramic probe approximately 2 feet long with an OD of 1/4" approximately 50 feet of 316 stainless steel tubing, 1/4 OD, approximately 2 feet of glass 'sock', and several size of air-vac pumps.

2. Plug the sample inlet port on the air-vac pump. Hook the air inlet port up to a tank of air and the outlet port to a Singer dry gas meter.

3. Measure the flow of air through each pump at tank pressure of 10, 20, 30 and 40 psig.

4. Attach approximately 20 feet of the stainless steel to the pump inlet. Attach the 2 foot ceramic probe to the other end of the stainless steel tubing. Cover the probe with the glass 'sock'. Repeat step 3.

5. Calculate the flow through the sample probe by subtracting the results obtained in Step 3 from that obtained in step 4.

6. Pick a cylinder pressure that will closely approximate the air and blend gas flows used in the flame optimization phase. Calibrate the pumps around these flows.

2.1.4 Miscellaneous Preparations

Additional modifications of instrumentation which were performed are described below.

1. Replumb the oxidant port to the burner head in stainless steel.

2. Place a sample introduction port with a cut-off valve in the oxidant line.

3. Prepare a wind-shield for the burner-head chamber to protect the flame from air flows at the sampling location.

2.2 Task 2 -- On-Site Analysis

The on-site work to be performed under Task 2 includes: instrumental set-up and verification check up at the sampling site; development of a procedure for the delivery of sample gas to the instrument on-site data collection and the collection of sample line runs for off-site analyses.

2.2.1 Instrument Set-Up and Check Out

Follow the procedure given in Section 2.1.1 for flame optimization and calibration.

2.2.2 Delivery of Sample Gas

A ceramic probe covered by a glass 'sock' will be inserted into the sample port. The remainder of the sample line will be constructed of heat traced 316 stainless steel tubing. The stainless steel will run from the sample port, out the side of the incinerator trailer, and down to the instrument.

The primary furnace off-gas will be at a negative pressure relative to atmosphere. The air used as the oxidant for the flame in the AA will be delivered through an air-vac aspirator. This will provide a vacuum on the sample gas line and draw the sample gas to the instrument. The sample system to be used for the primary furnace off gas is presented in Figure 1.

The sample line will have three distinct temperature zones as shown in Figure 1. The first zone is from the primary furnace sample port to approximately 1 foot before the air-vac pump. Temperature zone one will be maintained at temperatures in excess of 1200°F. Temperature zone two will begin at cool down so that the pump temperature will be less than 800°F (maximum temperature rating). The sample gas stream will be cooled to approximately 400°F for introduction into the burner head (temperature zone 3).

2.2.3 Collection of Sample Line Rinse

The elevated temperature of the sample line will minimize the loss of lead on the walls of the sample line. To evaluate the loss of lead in the cooler temperature zones, the section of tubing defined as temperature zones 2 and 3 will be removed at the end of each days' data collection and rinsed with dilute nitric acid and water. These rinses will be analyzed for lead.

2.2.4 On-Site Data Collection

The procedure for on-site data collection is described below.

1. Set up the instrument as described in Section 2.2.1.
2. Calibrate in the air-blend-acetylene flame mixture using 2, 5, and 10 mg/L standards. A laboratory QC of 8 mg/L will be analyzed to verify standards.
3. Set the pressure on the regulator of the auxiliary air being used to draw the primary furnace off gas down the sample line. The pressure for each pump was determined in Section 2.1.3.
4. Switch the valve on the oxident line to introduce the sample gas. Record the time of the valve switch. Let equilibrate.
5. Do not zero the baseline. Calibrate at least two times.
6. Turn the valve to return to the air-blend mixture and remove pressure from the auxiliary air tank. Record the time of the valve switch.
7. Use the methods of standard additions to determine the concentration of lead in the primary furnace off gas. (See Section 2.1.2.)

8. Record the temperature of temperature zones 1, 2, and 3 instrument operating parameters, nebulization rate, ambient temperature and pressure.

9. Repeat steps 3-9 at 20 minute intervals. (Reduce to ten minute intervals if possible.)

10. QC audit samples should be analyzed three times a day.

2.3 Task 3 -- Off-Site Analysis

Under Task 3, the tubing rinses collected during the on-site sampling and analysis efforts will be analyzed for lead using EPA Method 7740.

2.4 Task 4 -- Data Evaluation and Reporting

The data will be evaluated, and a final report prepared under Task 4. This will include calculations for vapor phase lead concentrations, as well as QA/QC results from all analyses. This data will be used to generate a distribution of vapor phase lead concentration versus time. The vapor phase lead results will also be correlated to the particulate loading and the temperature measured at the stack. The final report is currently scheduled for submittal in December 1987.

3.0 QUALITY ASSURANCE AND QUALITY CONTROL (QA/QC)

The objectives of the quality assurance program is to assure, assess and document the precision, accuracy and completeness of the data developed during this program. The sampling quality control will be described in Section 3.1. Analytical quality control, sample custody, performance and systems audits, data quality indicators and quality assurance reporting will be described in Sections 3.2 through 3.7, respectively.

3.1 Sampling Quality Control

Sampling equipment calibration and field sampling quality control are discussed below.

3.1.1 Sampling Equipment Calibration Procedures

The checkout and calibration of sampling equipment is an important function in maintaining data quality. Standard calibration procedures, as described in the EPA Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods (EPA 600/4-77-027b), are followed for all applicable equipment. Calibration requirements can also be found in the Texas Air Control Board Sampling Procedures Manual. If a referenced calibration technique for a particular piece of apparatus is not available, then a state-of-the-art technique will be used. A discussion of the techniques used to calibrate thermocouple temperature sensors and dry gas meters are discussed in Section 4.5 of Quality Assurance Project Plan for the Shirco Portable Pilot Test Unit Demonstration at Rose Township (QAPP).

The air, acetylene, blend, and standard gas rotameters will be calibrated in triplicate prior to sampling using a Teledyne-Hastings bubblemeter. These values will be corrected for temperature and pressure.

The air-vac pumps used to draw the primary furnace off-gas to the AA will be calibrated as discussed in Section 2.3.1 of this Test Plan.

3.1.2 Field Sampling Quality Control

The field sample quality control will include the collection of field blanks for the sample gas tubing rinses. The number of field blanks are based on the number of samples to be collected. A minimum of 10% field blanks will be collected in cases where more than ten samples are collected. The field blanks will be prepared by issuing the deionized water, the acid used for sample collection and preservation of the sample tubing rinses. These samples are then handled exactly like field samples. Field blanks provide information on background levels of the compounds of interest which may be present due to contamination of the sampling equipment.

3.2 Analytical Quality Control

Analytical quality control can be divided into two sections; the calibration of the analytical system and the quality control procedures used during analysis. These aspects of analytical quality control will be discussed in Sections 3.2.1 and 3.2.2, respectively.

3.2.1 Analytical Calibration

The most common type of calibration for analysis is the multipoint calibration, which will be periodically checked by calibration check samples, quality control check samples, etc. The multipoint calibration establishes the working range of the instrument or method. The concentration chosen should bracket the expected concentration of the analyte in the field sample. If the concentration of the analyte falls outside the established calibration curve, then the curve can be extended with additional standards or, preferably, the sample can be diluted until the analyte is within the working curve.

The data set generated by the multipoint instrument calibration will consist of pairs of values for the calibration concentrations (plotted as X_i) and instrument responses (Y_i). These data pairs are plotted on arithmetic graph paper and inspected visually for linearity. The measured net signal, S, should be a linear function, g(C), of the analyte concentration, C, i.e.,

$$S = g(C),$$

The equation of the resulting straight line can be expressed as

$$S = m\,C + b,$$

where m is the slope of the line and b the intercept on the S axis.

After the calibration curve is plotted and observed to be linear, the correlation coefficient, slope, and intercept are calculated, using the method of least squares linear regression analysis. The resulting equation is used to calculate the concentrations of the analyte in the samples analyzed.

3.2.2 Quality Control During Analysis

Those procedures which serve both control and accuracy assessment functions include:

- Replicate and repeat analyses;
- Field and reagent/laboratory blank analyses;
- Analysis of replicate samples;
- Control sample analyses;
- Spiked sample analyses; and
- Drift checks.

Analytical quality control functions include reagent blanks, replicate analyses, analysis of control samples and analysis of spiked samples. Extrapolation of the QC data to actual samples and measurements provides the mechanism by which error limits for various measurements may be estimated and the confidence in the measurement data defined.

A summary by parameter of the analytical quality control procedures is presented in Table 1. The quality control procedures for the on-site analyses are composed of instrument blanks and replicate analyses. A control standard from a source different from the one used for calibration will be used as a check of the calibration curve. Agreement must be within ±10% of the calibration curve to be acceptable. The off-site analysis quality control procedures will also include the analysis of digestion blanks, duplicate digestates, sample spikes and a field blank.

3.3 Sample Custody

See Section 4.6 of the Rose Township Demonstration Tasks QAPP.

3.4 Performance and Systems Audits

The purpose of a quality assurance audit is to provide an objective, independent assessment of a measurement effort. The ensure that the laboratory's data generating, data gathering, and measurement activities produce reliable and useful results. Cases occur where inadequacies are identified in the measurement system and/or the researcher's quality assurance program. In such cases, audits provide the mechanism for implementing corrective action.

A performance audit will be performed to assess the accuracy of the instrument gathering data. The vapor phase lead performance audit will include submission of standard solutions (reference standards) for off-site analyses. Reference standards will be submitted with the field samples. Such audit samples will be assigned a field number and will not be identified as audit samples.

These performance evaluation audits will answer questions regarding the following:

TABLE 1. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES GASEOUS AND AQUEOUS

Parameter	Analytical Method	Quality Control Check	Frequency	Acceptance Criteria	Action or Corrective Action
Pb	Atomic Absorption	Calibration blank	Prior to sample analyses	Blank less than two times detection limit	Repeat analysis
		Digestion blank	Prior to analyses	Less than three times element detection limit	Determine source of contamination and redigest
		Control sample	1) Immediately following calibration 2) 10% (minimum 2) thereafter	Measured value within ±15% of true value	1) Repeat analysis 2) Repeat calibration
		Mid-level standard	1 in 20 if more than 20 samples analyzed	Response within ±10% of original calibration curve	Repeat calibration
		Duplicate digestion and analysis	10% (minimum 1)	Relative percent difference (RPD) ≤20% for replicate determinations for quantitations greater than five times detection limit	Repeat duplicate analyses
		Sample spikes	10% (minimum 1)	Spike recovery between 70-130%	Repeat spike analysis
		Field blank	Minimum 1	None	Will be used to determine sources of contamination

- Accuracy and precision of the measurement system; and

- The quality control data as compared to the actual data collected.

3.5 Data Quality Indicators

See Section 9 of the Rose Township Demonstration Tests QAPP.

3.6 Corrective Action

See Section 10 of the Rose Township Demonstration Tests QAPP.

3.7 Quality Assurance Reporting

See Section 11 of the Rose Township Demonstration Tests QAPP.

4.0 PROJECT SAFETY

See Section 12 of the Rose Township Demonstration Tasks QAPP.

APPENDIX F

MONITORING STUDIES FOR VAPOR PHASE LEAD EMISSIONS FROM THE SHIRCO PORTABLE PILOT UNIT DURING THE ROSE TOWNSHIP DEMONSTRATION TESTS

RADIAN
CORPORATION

DCN 88 274-013-01

MONITORING STUDIES FOR VAPOR PHASE LEAD EMISSONS FROM THE SHIRCO PORTABLE PILOT UNIT DURING THE ROSE TOWNSHIP DEMONSTRATION TESTS

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1.0 INTRODUCTION

Radian Corporation, under contract to Enviresponse, Inc., conducted a comprehensive characterization of Shirco Infrared Systems' Portable Pilot Test Unit. The program was performed under the auspices of EPA's Superfund Innovative Technology Evaluation (SITE) program at the Rose Township site near Flint, Michigan. The site was known to be contaminated with PCBs and lead.

Many of the environmental issues relating to the incineration of hazardous wastes or contaminated materials have revolved around the destruction of organic constituents. However, recently there has been a growing awareness concerning the atmospheric emissions of metals associated with the organically contaminated materials.

At the Rose Township site, as with many others, metals and PCBs were the main environmental concerns. The maximum concentration of lead in the soil at Rose Township was approximately 1,500 ug/g (ppm). Experience at another site using Shirco technology (Peak Oil) indicated that lead was vaporized from the soil in the primary furnace. It later recondensed to form very fine particles which were rather poorly removed by a wet scrubber.

Radian Corporation, through work with the Electric Power Research Institute and the Department of Energy, has developed a continuous vapor phase monitor for selected metals, including lead. The system has the capability of providing "real time" data and can be used for process evaluation and control. This monitor was used during the Rose Township program in an attempt to better evaluate the fate of lead during incineration of the contaminated soil. The intent was that these data could be used to evaluate the volatilization of lead in the primary chamber while varying the incinerator operating over a range of conditions required to destroy the organic material. However, in this particular application, essentially no vaporous lead was observed in the primary off-gases, and results are therefore reported at or less-than-detection-limit values.

2.0 EXPERIMENTAL

Before going to the field, a detailed test plan was prepared and submitted. Radian's on-line, continuous vapor phase trace element monitor for the primary furnace off-gas was adapted to this particular usage. A vapor phase lead standard (tetraethyl lead) was developed so that calibration of the system in the field was simplified to injection of aqueous standards. In addition, the sampling location was chosen, the sampling methods refined, and the protocol for on-site data collection was chosen. All of these details were set out in the submitted test plan; the interested reader is referred to that document for more specific information. ("Test Plan for the Monitoring of Vapor Phase Lead Emissions from the Shirco Portable Pilot Unit During the Rose Township Demonstration Tests," D.L. Bisson, Radian Corpoartion, October 30, 1987.)

Once on site, the preliminary work included: instrumental set-up and basic operation established; development of a delivery system to get the sample gas to the instrument; verification of the on-site data collection system; and construction of an environmental shelter around the instrument and delivery system.

Experiments were performed using the GBC-900 atomic absorption spectrophotometer. A single-element hollow cathode lamp was used for the determination of lead. The GBC was equipped with an impact bead nebulizer and pre-mix air-acetylene burner head. Data were recorded in two formats: numerical output from the integration mode of the GBC-900, and direct output to a Soltex 1242 strip chart recorder. Industrial grade air and acetylene were used to establish the flame. The blend gas originally chosen had a certified composition of 5.0 percent CO_2, 8.0 percent O_2, and 87 percent N_2.

The primary furnace off-gas sampling system was developed at Radian and modified in the field to deliver a representative sample. The probe consisted of a 316 stainless tube covered by a glass 'sock' inserted into the sample port. The remainder of the sample line was heat traced 316 stainless steel tubing. The stainless steel ran from the sample port, out the side of the incinerator trailer, down to the heated sample pump and then to the instrument, a total run of about 20 feet.

The sample line had distinct temperature zones. The first zone extended from the primary furnace sample port to approximately three feet before the vacuum pump. In this zone, the temperature was always in excess of 750°F. Since the pump was rated at 700°F, the gas temperature was maintained at approximately 700°F through the pump. Finally, the sample gas stream was cooled to approximately 480°F for introduction into the burner head.

The primary furnace off-gas was at a negative pressure relative to atmosphere. Therefore, a heated vacuum pump was used to deliver the primary furnace off-gas to the fuel inlet. (Laboratory tests had earlier indicated that sample introduction to the oxidant side negatively influenced nebulization rate and produced back-pressures in the sample line.) To prevent changes in the flame characteristics from one type of sample to another, all gases (calibration, blend, sample) were introduced into the fuel side feed of the burner head.

A difficulty was encountered with the composition of the blend gas. It was chosen to approximate the gas composition (from Shirco data) expected in the primary off-gas (i.e., about 5% CO_2 and 8% O_2). In fact, the primary gas was 4% CO_2 and 18% O_2. With the resources available on site, the best alternative was to use air (21% O_2) as the blend gas. Before introduction it was heated to 480°F. With this adaptation, the flame characteristics remained nearly constant during both calibration and sampling.

The operational procedure followed on-site was:

- Calibrate in the air-ambient air-acetylene flame mixture using 2, 4, 8, and 10 mg/L standards. A laboratory QC sample of 5 mg/L was analyzed to verify standards.

- Set and calibrate the primary furnace off-gas and ambient air sampling pumps to deliver similar gas volumes using a dry gas meter.

- Carry out multi-point calibrations at least two times.

- Use the methods of standard additions to determine the concentration of lead in the primary furnace off gas.

- Record the temperature of temperature zones 1, 2, and 3 instrument operating parameters, nebulization rate, ambient temperature and pressure.

- Analyze QC audit samples at least two times a day.

- The minimum sampling intervals were approximately 30 minutes.

Experience has shown that lead does not deposit in the hot zone of the sample line. However, the part of the line below 750°F was washed out at the end of each day and the rinse retained for later analysis (for lead, EPA Method 7740).

3.0 RESULTS AND CONCLUSIONS

The vapor-phase, real-time lead detection system was up and operating properly for the three days of the SITE testing on the Shirco Infrared Incinerator at the Rose Township site. During this period, 14 valid runs were made. On two of the runs there was a slight response; on the other twelve runs, nothing was seen. The results are summarized in Table 3-1.

The two slight responses refer to two cases where there appeared to be a signal above the background noise level, but it was less than three times the noise level -- the criterion usually used to define the limit of quantification. These two faint signals were assigned the value at the detection limit in Table 3-1. Thus, we may report that in two of fourteen tests, lead level near 3 ppbv were detected.

At the end of each day of testing, the sample delivery system was broken down and the section where the temperature fell below 750°F was washed out to capture any retained particulate material. Some lead was detected in the rinse water; see Table 3-2. However, these data indicate that the lead deposition from the flue gas was also in the low ppb range. We feel these values are not very accurate (no formal protocol exists), and they serve only to indicate that large quantities of lead were not depositing in the sample delivery lines.

Thus, after three days where the system appeared to be working well (calibrations and QC samples repeated satisfactorily), there were only two minor indications of lead and twelve results lower than the detection limit of 2.7 ppbv (equivalent to about 0.00025 gr/dscf). Radian, in consultation with Enviresponse and the EPA project manager (Howard Wall), decided not to continue the testing since no condition during the following week of testing was going to be as severe (temperature and residence times) as the conditions maintained during the first three days of SITE testing.

TABLE 3-1. VAPOR PHASE LEAD CONCENTRATIONS IN THE PRIMARY FURANCE OFF-GAS DURING THE COMPREHENSIVE TEST OF THE SHIRCO INCINERATOR AT ROSE TOWNSHIP, MI

Date	Time	Run	ppbv Pb[a]
11-3-87	1528	1	<2.7
	1551	2	<2.7
	1613	3	<2.7
	1646	4	<2.7
11-4-87	1736	1	2.7
	1838	2	<2.7
	1934	3	<2.7
11-5-87	1035	1	<2.7
	1108	2	2.7
	1325	3	<2.7
	1442	4	<2.7
	1515	5	<2.7
	1547	6	<2.7
	1614	7	<2.7

[a] Vapor phase lead concentration with the liquid phase to vapor phase correction factor applied.

TABLE 3-2. VAPOR PHASE LEAD CONCENTRATIONS IN THE PRIMARY FURNACE OFF-GAS SAMPLE ACQUISITION SYSTEM

Date	Liquid Phase Pb Concentration ug/mL	Rinse Volume mL	Sample Time minutes	Sample Flow LPM	ppbvPb[a]
11-3-87	0.14	64.8	150	6.705	<2.7
11-4-87	0.090	112.9	72	6.006	3.4
11-5-87	0.31	147.4	174	7.081	5.4

[a] Vapor phase lead concentration with the liquid phase to vapor phase correction factor applied.

Therefore, even when the uncertain but highest values for the delivery system wash and detector results were combined, vapor phase lead levels measured in the primary off-gas never reached 10 ppbm (equivalent to less than 0.001 gr/dscf). Indeed, most measurements indicate that the vapor phase lead was usually less than one-quarter of this level.

4.0 QUALITY CONTROL

This section goes into some detail concerning the QA/QC procedures followed in this study. The level of detail is greater because most of these items were not covered in the test plan. It is intended for the reader interested in the performance charcteristics of the lead monitor and will serve to document the actual procedures.

4.1 Documentation

4.1.1 Sample Labeling System

The vapor phase lead data were recorded by a strip chart recorder. The experimental parameters such as date, time, gas flows, standard concentrations, and other experimental parameters were recorded on the recorder paper. The instrumental integration of the sample absorbance by the Buck AAS were also recorded on the recorder paper alongside the corresponding chart data. The data were then reduced and transferred to the laboratory notebook. The recorder paper was placed in a hard data file.

4.1.2 Laboratory Notebook

The laboratory notebook is a bound, paginated notebook. The lab book number, project name, project dates, and person responsible for recording data are presented on the first page.

Entries include general sampling and analytical information with sufficient detail to reconstruct the sampling and analytical activities without reliance on memory. All entries are in ink, or computer printouts, with no erasures. Incorrect entries are crossed out with a single strike and initialed. Each page is dated and signed by the person making the entry.

4.2 Data Validation

All measurement data were validated for representative conditions during sampling and analysis, for acceptable sample collection procedures, for consistency with expected results, and for adherence to the prescribed analytical procedure.

Several of the data validation criteria involve specific calculations. These are presented below.

4.2.1 Instrument Calibration

The data set generated by the multipoint instrument calibration consisted of pairs of values for the calibration concentrations (plotted as X_i) and absorbance (Y_i). These data pairs were plotted on arithmetic graph paper and inspected visually for linearity. The measured signal, Y, should have been a linear function of the analyte concentration, X. That is,

$$Y_i = mX_i + b$$

where m was the slope of the line and b the intercept on the y axis.

After the calibration curve was plotted and observed to be reasonably linear, the correlation coefficient, slope, Y-intercept and X-intercept were calculated, using the method of least squares linear regression analysis. The resulting equation was used to calculate the concentrations of the analyte in the samples analyzed.

4.2.2 Precision

Control limits for control sample analyses and acceptability limits for replicate analyses are based upon precision and given in terms of the coefficient of variation CV (i.e., the relative standard deviation). The standard deviation of a sample set is calculated as:

$$S = \text{standard deviation} = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$

where:

x_i = individual measurement;

$\bar{x}$ = mean value for the individual measurements; and

n = number of measurements.

The CV, expressed as percent, is then calculated as:

$$CV = (S/\bar{x}) \times 100.$$

4.2.3 Percent Recovery

The percent recovery of the quality control standard was calculated using the following equation:

$$\% \text{ REC} = \frac{EC}{TC} \times 100$$

where:

% REC = percent recovery;

EC = experimental concentration determined from the instrument response and the calibration curve;

TC = true concentration; and

100 = converts from decimal to percent.

4.3 Analytical Quality Control

A summary of the quality control procedures for this method is given in Table 4-1. Table 4-2 summarizes the quality control data obtained for the atomic absorption analysis of lead.

4.4 Analytical Quality Assurance

A set of three audit samples were analyzed daily during the field test. These results were submitted to Radian's quality assurance group. The results of the Rose Township Performance Evaluation are summarized in Table 4-3.

The recovery for lead sample 1087-24 was within the normal expected range of recovery (100 ± 5%) for this type of analysis. Although the recoveries for sample 1087-25 and 2087-26 were not within normally acceptable accuracy objectives, these samples were spiked at levels less than the quantification limit of five times the detection limit of 0.1 milligrams per liter of lead. It should be noted that audit values less than five times the detection limit are not indicative of typical performance.

[illegible]

Parameter	Analytical Method	Quality Control Check	Frequency	Acceptance Criteria	Action or Corrective Action
Pb	Atomic Absorption	Calibration blank	Prior to sample analyses	Blank less than two times detection limit	Repeat analysis
		Digestion blank[a]	Prior to analyses	Less than three times element detection limit	Determine source of contamination and redigest
		Control sample	1) Immediately following calibration 2) 10% (minimum 2) thereafter	Measured value within ±15% of true value	1) Repeat analysis 2) Repeat calibration
		Mid-level standard	1 in 20 if more than 20 samples analyzed	Response within ±10% of original calibration curve	Repeat calibration
		Duplicate digestion and analysis[a]	10% (minimum 1)	Relative percent difference (RPD) ≤20% for replicate determinations for quantitations greater than five times detection limit	Repeat duplicate analyses
		Sample spikes[a]	10% (minimum 1)	Spike recovery between 70-130%	Repeat spike analysis
		Field blank[a]	Minimum 1	None	Will be used to determine sources of contamination

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[a] For aqueous sample analysis only.

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TABLE 4-2. QUALITY CONTROL DATA FOR THE ATOMIC ABSORPTION ANALYSIS OF LEAD IN THE PRIMARY FURNACE OFF-GAS

Sample Date	Correlation Coefficient	Control Standard (% Recovery)
11-2-87	0.999	94.
11-3-87	0.998	99.
	0.998	94
	0.999	100
	0.999	94
11-4-87	0.999	112
	0.998	101
	0.999	96
11-5-87	0.999	90
	0.999	89
	0.999	102
	0.996	100
	0.998	91
	0.999	97
	0.999	94

TABLE 4-3. OCTOBER 1987 PERFORMANCE AUDIT RESULTS FOR LEAD IN WATER SAMPLES (FIELD TEST - ROSE TOWNSHIP, MI)

	Parameter-Pb
Method Detection Limit[a] (mg/L)	0.1
1087-24	
Certified Value (mg/L)	4.0
Reported Value (mg/L)	4.02
Percent Recovery[b] (%)	101
1087-25	
Certified Value (mg/L)	0.2
Reported Value (mg/L)	0.134
Percent Recovery[b] (%)	67
1087-26	
Certified Value (mg/L)	0.1
Reported Value (mg/L)	0.046
Percent Recovery[b] (%)	46

[a] Reported detection limit.

[b] Sample Recovery (%) = $\frac{\text{Reported Value}}{\text{Audit Value}}$ x 100.

NS - Not Specified.

VPTE - Vapor Phase Trace Element.

APPENDIX G

EVALUATION OF EP AND TCLP EXTRACTION PROCEDURES FOR Pb-CONTAMINATED SOILS AND COMBUSTION RESIDUES

EVALUATION OF EP AND TCLP EXTRACTION PROCEDURES FOR PB-CONTAMINATED SOILS AND COMBUSTION RESIDUES

1.0 INTRODUCTION

Results from two recent SITE soil incineration programs involving Pb-contaminated soils have indicated that Pb concentrations were generally greater in EP extracts than in comparable TCLP extracts (Reference SITE reports from Peak Oil and Rose Township). Based on limited supportive evidence from these projects, the reasons for these results were unclear. Therefore, a more intensive investigation was conducted on five selected samples from these SITE projects. The objective of this investigation was to compare Pb concentrations in EP and TCLP extracts performed on identical samples on the basis of differences in final pH of the extracts and in total alkalinity of the solid material.

2.0 METHODS

Five waste materials were selected to represent each soil-incineration process stream from the Peak Oil (PO) and Rose Township (RT) SITE programs. Samples included furnace ash and waste feed from each of the programs and a sample of scrubber sludge from the Peak Oil program (Table 1). Each waste sample was extracted using the EP toxicity and TCLP procedures. The final pH of the extract was recorded for both methods after the extraction was completed. The pH of one sample extracted with the EP was continuously monitored by strip-chart recorder while acid was added with an autotitrator. The strip-chart record confirmed that the pH of the extraction was maintained between 4.8 and 5.0.

Two samples, waste feed and scrubber sludge from Peak Oil, required extraction with fluid number two of the TCLP. This requirement was based on the results of the procedure for determination of the appropriate extraction fluid.

Total alkalinity was determined by titrating a 5.0-gram sample with 1.0N HCl to a pH of 4.5. Lead (Pb) concentrations in extracts were determined by graphite furnace atomic absorption spectrophotometery (GFAAS).

3.0 RESULTS

Final pH values and Pb concentrations for each extraction procedure are reported in Table 1. The greatest Pb concentration found in this group of samples was 46 ppm in the EP extract of the PO-FA sample. The corresponding Pb concentration in the TCLP extract was 0.84 ppm. The final pH values of the EP and TCLP extracts for this sample were 5.0 and 7.8, respectively. The association between the final pH of EP and TCLP extracts and Pb concentrations is illustrated in Figure 1. The higher final pH of the TCLP apparently reduced the solubility of Pb in the extract relative to that in the EP extract.

A higher Pb concentration (35 ppm) was found in the TCLP extract than in the EP (25 ppm) extract for the PO-WF sample. Corresponding pH values were 4.4 and 4.8 in the TCLP and EP extracts, respectively. The lower pH in the TCLP extract resulted from using extraction fluid number 2.

A consistent inverse relationship between Pb concentration and final pH of EP and TCLP extracts was found for the remaining samples analyzed (Figure 1). However, Pb concentrations were all below 5.0 ppm in all of the extracts of the remaining samples.

Lead concentrations in EP and TCLP extracts were clearly unrelated to total Pb found in the solid samples. For example, the PO-SS sample contained the highest Pb concentration while the Pb concentrations in EP and TCLP extracts were lowest among the samples tested. The RT-WF sample containing the lowest Pb concentration produced EP and TCLP extracts containing Pb concentrations of 3.0 and 1.4 ppm, respectively. These disparities between total and EP and TCLP extractable Pb levels are most likely due to different Pb solid phases in the samples which control Pb solubility in the extracts.

TABLE 1. ANALYSIS OF EP AND TCLP EXTRACTS OF ASH SAMPLES FROM PEAK OIL AND ROSE TOWNSHIP SITE PROGRAMS

Sample Identification	Date	Time	Total Pb (ug/g)	Total Alkalinity (mmole/g)	Final pH	Volume Acid Added (ml)	Pb Conc. (ug/mL)	Final pH	Pb Conc. (ug/mL)	Extraction Fluid No.
PO - FA	8/2/87	Comp.	6000	0.4	5.0	200	46 (28)[a]	7.8	0.84 (0.008)[a]	1
PO - WF	8/2/87	Comp.	4900	0.12	4.8	157	25 (29)[a]	4.4	35 (2.5)[a]	2
RT - FA	11/11/87	1450	1200	0.18	5.0	70	4.9 (0.06)[b]	5.6	2.8 (4.1)[b]	1
RT - WF	11/10/87	1245	500	0.07	5.0	115	3.0 (0.07)[b]	5.4	1.4 (0.55)[b]	1
PO - SS	8/2/87	Comp.	90000	1.4	5.0	200	0.29 (40)[a]	5.2	0.23 (0.26)[a]	2

PO - Peak Oil

RT - Rose Township

FA - Furnace Ash

WF - Waste Feed

SS - Scrubber Sludge

[a] Comparable result from Peak Oil Report

[b] Comparable result from Rose Township Report

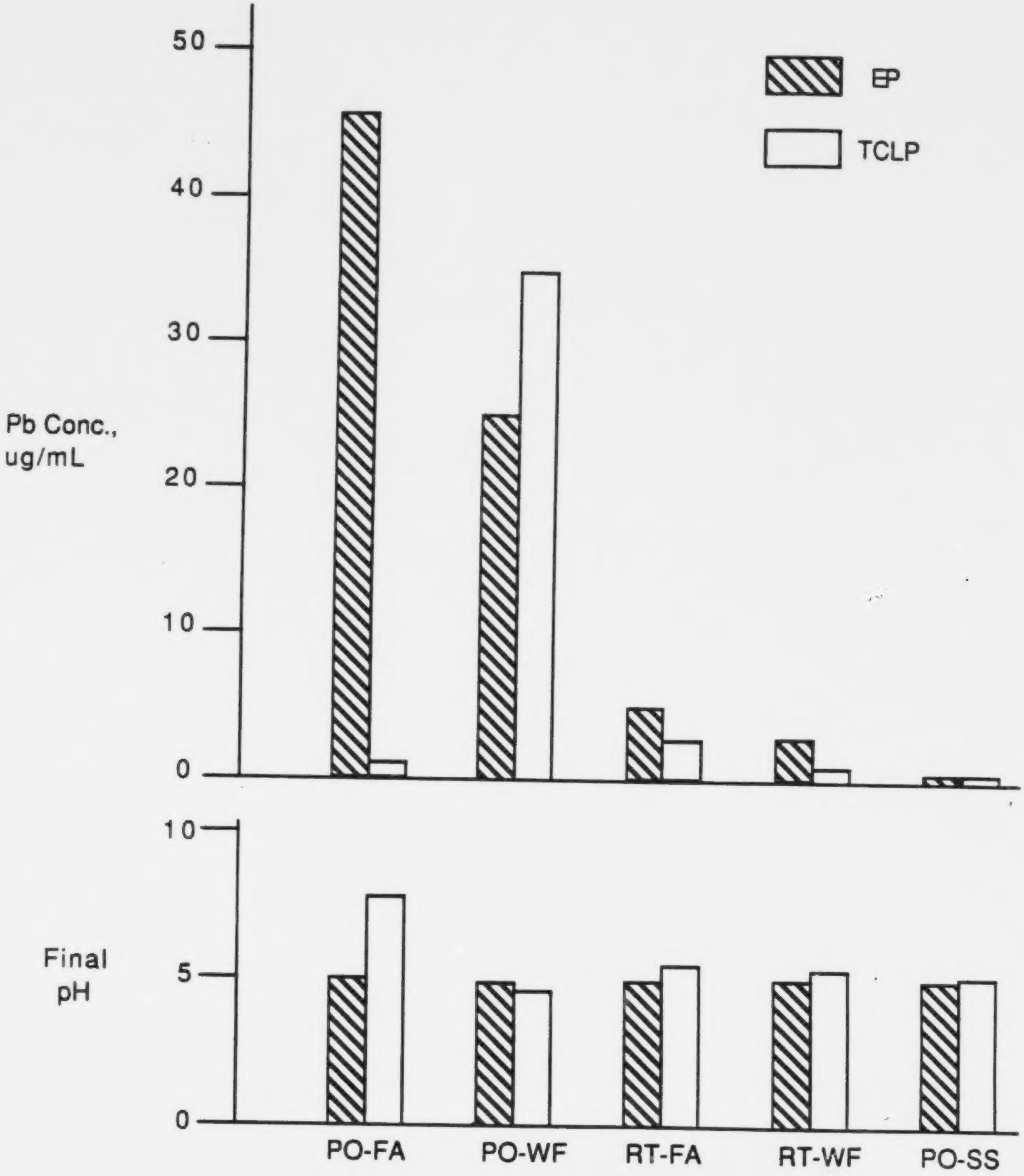

Figure 1. Pb concentrations and Final pH values for EP and TCLP extracts